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Pratt & Whitney Aircraft DIVISION OF UNITED AIRCRAFT CORPORATION

EAST HARTFORD 8, CONNECTICUT

Annual Progress Report
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Technical Management: National Aeronautics & Space Administration, Lewis Research Center, Nuclear Technology Branch, Herman Schwartz

Approved by:

R. C. Huttinger
Program Manager

W. J. Lueckel, Chief,
Space Power Systems

Pratt & Whitney Aircraft DIVISION OF UNITED AIRCRAFT CORPORATION

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NOTICE

FOREWORD

This report describes the research activity carried out in fulfillment of Contract NASw-104 as modified by Amendments 1 through 9 during the period from January 1 through December 31, 1963.

ABSTRACT

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The program for the year was divided into four parts, each of which is reported in separate sections. An additional section describes data obtained in 1962 and to date unreported because of rig difficulties during that period.

A screening rig was constructed to enable evaluation of coating behavior at elevated temperatures so that coating volatilization in the emittance rigs could be avoided. Three materials have been tested to date, namely, Kennametals K-151A and K-162B, and iron titanate. The Kennametals were found to be stable up to 1600°F and the iron titanate up to 2000°F.

Emittance values were obtained for several materials and were compared with previously obtained values to determine reproducibility. These materials included crystalline boron, oxidized Kennametals K-151A and K-162B, calcium and iron titanates, nickel-chrome spinel, and silicon carbide. Data from all of these specimens were in good agreement with that obtained previously except for that of the crystalline boron (which was slightly lower), one of the nickel-chrome spinel coatings (which was slightly higher), and one of the calcium titanate coatings (for which the emittance behaviour was irregular). In addition, tests were run on a spinel enamel and on AISI-310 stainless steel and tantalum to evaluate the operation of the total hemispherical emittance rig.

Tests of four coated radiator segments exposed to temperatures between 650 and 700°F and vacuum for periods ranging from 12,700 to 15,000 hours were completed and a post test analysis was made. Analysis included mechanical, metallographic, and X-ray diffraction testing as well as residual gas analyses and vacuum chamber leak testing. The analyses confirmed that, in general, the segments withstood the extended exposure to high temperature and vacuum satisfactorily.

Methods for applying silicon carbide coatings by aluminum phosphate bonding were investigated, but, although some improvement was realized, a satisfactory technique has not been developed.

Difficulties encountered with the total hemispherical emittance rig in the latter part of 1962 were resolved and emittance data obtained during this period are now reported. Materials included are Hastelloys C and X, oxidized Kennametals K-151A and K-162B, titanates of barium, calcium, iron, iron with alumina, and strontium, and silicon carbide. Author

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I. VOLATILIZATION TESTS

A test rig has been constructed to provide preliminary information on the behavior of coatings subjected to temperature and high vacuum conditions. Testing of selected coatings in this apparatus will establish the maximum temperatures to which these coatings may be heated before measurable volatilization occurs. This information can be utilized to prevent overheating of coatings in the emittance rigs and the attendant plating of volatilized material on the electrical circuitry.

A standard glass bell jar 15 inches high and 15 inches in diameter was used for the vacuum chamber to permit observation of the specimen during testing and to simplify cleaning after testing. The bell jar was seated on a special base plate equipped with a cooled radiation shield to absorb heat radiated by the specimen and to prevent the bell jar from heating during testing. Vacuum grease was used to seal the bell jar to the base plate and a metal shatter guard was placed over the bell jar to protect personnel in the event of bell jar failure. Provision was made for mounting a 35 mm camera on the bell jar shield to permit the specimen to be photographed. An external view of the rig is shown in Figure 1.

An instrumentation flange (shown in Figure 2) supported the specimen and provided feedthroughs for power and thermocouple leads. Terminals were provided for three platinum-platinum 10 per cent rhodium, two chromel-alumel, and two tungsten-tungsten 26 per cent rhenium thermocouples. A one-inch square sheet of stainless steel was suspended from the upper specimen support about two inches from the specimen to facilitate observation of any deposition which might occur.

The evacuation system consisted of a 400 liter-per-second oil diffusion pump backed by a 13 cubic-foot-per-minute mechanical roughing pump and a liquid nitrogen cold trap. Pressures as low as 3.0×10^{-6} mm Hg were obtained with the specimens heated to temperatures of 1600°F. Pressure measurement was made with a Bayard-Alpert type ionization gage.

To date, coatings of oxidized Kennametals K-151A and K-162B and an iron-titanate coating have been tested in the rig. Figure 3 and Tables 1 and 2 show that the two Kennametal coatings were stable up to 1600°F when exposed to a vacuum in the 10^{-6} mm Hg range. At temperatures above 1600°F, the coatings volatilized rapidly enough to measurably increase the pressure in the chamber. This increase was accompanied by the appearance of dark spots at various places on the specimens. These dark spots are indicative of regions of lower apparent temperature which may be a result of local coating separation. During testing of the Kennametal K-162B coating at 1700°F, it was observed that the thermocouple indications decreased at a constant power setting. Although this behavior may be a result of an emittance increase, it is more probable that the coating was volatilizing and introducing errors into the thermocouple readings.

As shown in Figure 3 and Table 3, the iron titanate coating was stable up to a temperature of 2000°F but volatilized rapidly when heated above 2150°F. At 2100°F the thermocouple indications decreased when the power setting remained constant for the same reasons described above.

II. EMITTANCE MEASUREMENTS

A. AISI-310 Stainless Steel

An AISI-310 stainless steel tube in the as-received condition was tested in the total hemispherical emittance rig in conjunction with the investigation of temperature measurement discrepancies discussed in Section V of this report. Emittance values were obtained between 1000 and 2000°F and appear in Table 4 and Figure 4. During the initial run, the emittance rose from 0.26 to 0.29 as the temperature increased from 1000°F to 2000°F. The specimen was retested at a later date and, as shown in Figure 4, the emittance values were lower than initially. Similar drops in emittance have been observed previously and have been attributed to surface clean-up resulting from exposure to elevated temperatures and reduced pressures.

B. Tantalum

Total hemispherical emittance measurements were made on two uncoated tantalum tubes to test the operation of the total hemispherical emittance rig. Three test runs were made with one tube, and a single test run was made with the second tube. All tests were conducted in the total hemispherical emittance rig. As shown in Table 5 and Figure 5, the data fall along two general curves with data from the first specimen comprising the lower curve, and that from the second specimen comprising the upper curve. The lower curve indicates a gradual increase in emittance from 0.14 at 900°F to 0.20 at 2200°F, whereas the upper curve indicates an emittance increase from 0.17 at 1000°F to 0.25 at 2200°F. The cause of this discrepancy in emittance between the two specimens is not known but may have been due to slight differences in surface roughness. The specimens were similar in appearance, and there were no visible changes as a result of testing.

C. Crystalline Boron

A coating of crystalline boron was plasma-arc sprayed onto a columbium - 1 per cent zirconium tube. Considerable difficulty was encountered in making the powder adhere to the substrate, and the resulting coating was less than 1 mil thick. The powder used had particle sizes ranging from 62 to 74 microns in diameter. The coating was gray, fairly hard, and had a matte texture finer than that of 320 grit emery cloth. The coating-substrate bond strength was excellent.

Total hemispherical emittance measurements were made between 300 and 2200°F, and data are presented in Table 6 and Figure 6. The emittance increased from 0.65 at 300°F to 0.74 at 1300°F and remained constant up to 1700°F. During the second heating cycle, the emittance obtained between 1500 and 1700°F was slightly lower, and with further heating to 1900°F the emittance increased to a maximum of 0.73 and then decreased to 0.70 at 2200°F. During cooling, the emittance remained at or below 0.70. The lowest emittance data previously reported (Technical Report PWA-2206) for crystalline boron was slightly higher than 0.74. It is possible that the lower values resulted from the thinness of the coating. No visible changes in the coating occurred as a result of testing.

D. Oxidized Kennametal K-151A

The specimen was prepared by plasma-arc spraying a 4-mil thick coating of oxidized Kennametal K-151A onto an AISI-310 stainless steel tube. (The material was oxidized by Kennametal, Incorporated by heating in air at 1600°F for 20 minutes.) The resulting coating was dark gray, fairly hard, and had a fair coating-to-substrate bond strength. The matte texture was similar to that of 240 grit emery cloth.

Emittance testing was conducted in the total hemispherical emittance rig at temperatures below 1600°F since testing in the volatilization rig indicated that the coating became unstable at higher temperatures. The emittance of the coating (see Table 7 and Figure 7) increased from 0.82 at 800°F to 0.88 at 1600°F and the same values were obtained during cooling. These values are in good agreement with the values based on thermocouple temperature measurements reported previously for this material in Pratt & Whitney Aircraft Report PWA-2206. The more recent values are somewhat higher, however, than the values obtained previously using optical pyrometer temperature values. At 1600°F the values recently obtained using optical pyrometer temperature measurements are in close agreement with values reported by Wade and Casey.[1]

E. Oxidized Kennametal K-162B

Emittance measurements of a coating of oxidized Kennametal K-162B were made to permit further comparison of Pratt & Whitney Aircraft emittance measurements with those of Wade and Casey[2] who measured the emittance of an oxidized sheet of Kennametal K-163B1. Unfortunately, K-163B1 is no longer available and therefore measurements were made on the replacement material, K-162B. The oxidized material was supplied in powder form with particle diameters between 53 and 88 microns. The powder was plasma-arc sprayed onto an AISI-310 stainless steel tube to form a 5-mil thick coating which was dark gray, fairly hard, and had a fair coating-to-substrate bond strength. The texture of the coating was similar to that of 240 grit emery cloth. No change in the coating characteristics occurred as a result of testing.

As was the case with Kennametal K-151A, emittance measurements were restricted to the temperature range of 800 to 1600°F. As shown in Table 8 and Figure 8, the emittance increased from 0.82 at 832°F to 0.89 at 1500°F. These values are lower than those obtained by Wade and Casey at temperatures below 1400°F, but, between 1400 and 1600°F, the values are in almost exact agreement.

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[1]Wade, W. R., and Casey, F. W., Jr., Measurement of Total Hemispherical Emissivity of Several Stably Oxidized Nickel-Titanium Carbide Cemented Hard Metals from 600° to 1600°F. NASA Memorandum, 5-13-59L, National Aeronautics & Space Administration, Langley Field, Virginia, June 1959.

[2]Ibid.

F. Calcium Titanate

Five calcium titanate coatings were tested. The first was aluminum phosphate bonded to a columbium - 1 per cent zirconium tube and was tested to evaluate the coating procedure used. The remaining coatings were plasma-arc sprayed onto columbium - 1 per cent zirconium tubes and were tested to evaluate the effects on emittance of exposure to elevated temperature.

First Specimen - As discussed in Section IV, an investigation of aluminum-phosphate bonding procedures was conducted to develop a curing procedure which would permit aluminum-phosphate bonded coatings to withstand exposure to elevated temperatures. A specimen prepared by the optimum procedure developed was tested to evaluate the effectiveness of the coating procedure.

Before testing, the coating was 4 mils thick, cream colored, and hard. The texture was similar to that of 320 grit emery cloth and the coating-to-substrate bond was good. Calcium titanate powder procured from Metco, Incorporated, was used in preparing the coating. The emittance data obtained (Table 9 and Figure 9) show a sharp decrease in emittance with increasing temperature. This is not typical of calcium titanate coatings and probably is caused by the aluminum-phosphate binder. Further, the values obtained between 300 and 500°F were significantly higher than 0.90 which is not representative of the material and probably reflects the effects of end losses.

Second Specimen - The second specimen had a light gray 5-mil thick coating. Emittance measurements were made between 300°F and 1600°F and results are presented in Table 10 and Figure 10. The emittance of the coating increased from 0.78 at 300°F to about 0.90 at 1300°F and remained at that level to 1600°F at which time the test was terminated. These values agree with those reported in Technical Report PWA-2206 for $CaO \cdot TiO_2$ and $SrO \cdot TiO_2$.

When the specimen first reached 1000°F, it was observed that the color of the chamber walls changed from gray to a brilliant blue. As may be seen in Table 10, the pressure level rose one decade at 900°F, remained at that level at 1000°F, and then returned to its original level at 1100°F. The pressure remained in the 10^{-7} mm Hg range until temperatures in excess of 1300°F were attained, and then it once again rose to the 10^{-6} mm Hg range. The chamber pressure was low enough during the period when the color changed that it was not believed that excessive volatilization had taken place, and termination of the test was not warranted since the instrumentation remained usable. Finally, it would not be expected that condensate from a calcium titanate coating would be blue. When the vacuum chamber was opened, no foreign material was found on the instrumentation flange although the chamber walls were still blue. The cause of this blue coating has not been determined. There were no changes in the appearance of the coating as a result of testing.

Third Specimen - The third specimen had a coating 2 mils thick which was light gray, and hard. The coating-to-substrate bond strength was good and the texture was similar to that of 320 grit emery cloth. As anticipated, the emittance rose to 0.90 at 1400°F with a value of 0.92 being obtained at 1500°F (see Table 11 and Figure 11). A high emittance level was maintained during cooling to 1100°F when the test was terminated. It is noted that the value obtained at 1500°F is about 1.6 per cent higher than that usually obtained with plasma-arc sprayed coatings of calcium titanate. The coating was somewhat darker after testing, but otherwise was unchanged in appearance.

Fourth Specimen - The fourth specimen had a 5-mil thick coating which was gray, hard, and had a good coating-to-substrate bond strength. The matte texture was similar to that of 180 grit emery cloth. Initial testing was conducted at 1000°F. During heating, the emittance never exceeded 0.82, but, after 25 hours at 1000°F, it rose to 0.87 where it remained for the next 100 hours (see Table 12 and Figures 12 and 13). During subsequent cooling and heating between 800 and 1100°F the higher emittance values were maintained. Continued heating at 1100°F caused the emittance to increase to 0.88. This value was also maintained during cooling and appears to be more stable than the value obtained at 1000°F. Endurance tests of approximately 24 hours each were conducted at 1200 and 1300°F, but no further emittance changes occurred. After 25 hours of testing at 1400°F, however, the emittance decreased from 0.88 to 0.86 (see Figure 14). The specimen was then heated to 1500°F to permit the use of an optical pyrometer for checking the thermocouple indications. As shown in Table 12, the optical pyrometer reading was 6°F lower than that of the thermocouple. This results in corresponding emittance values of 0.88 and 0.86 respectively. During cooling, the thermocouples continued to indicate an emittance of 0.86. The coating was unchanged as a result of testing.

Fifth Specimen - The coating on the final specimen contained blue and white particles on a light gray background and was tested between 300 and 1500°F. Results appear in Table 13 and Figures 15 through 17. The total hemispherical emittance of the coating was measured as the specimen was heated from 300 to 1000°F (see Figure 15). A 190-hour endurance test was conducted at 1000°F. As shown in Figure 16, the emittance rose slightly during the first 50 hours from 0.84 to 0.86, and then remained relatively constant throughout the last 140 hours. The specimen was then cooled to 600°F and then heated to 1100°F (see Figure 15). Successive endurance runs were made at 1100°F, 1200°F, 1300°F, and 1400°F with no change in the emittance noted. Most of the endurance tests were run for one day, but the test at 1300°F encompassed a weekend and covered a period of 118 hours. At 1500°F the emittance began to decrease and after 28 hours it had dropped from 0.86 to 0.72 (see Figure 17). After 28 hours at 1500°F the temperature of the specimen was decreased and the emittance remained at 0.72, thus indicating that a permanent change in the coating had occurred. The thermocouple data appears to be reliable since, at the end of the endurance test at 1500°F, thermocouple and optical pyrometer indications were in good agreement. After testing, the coating was uniformly gray. Similar color changes with calcium titanate specimens have occurred previously (see Technical Report PWA-2206, page 115).

Conclusions - The results from the fifth specimen were not as anticipated since the emittance of the specimen never attained 0.90 even at 1400 or 1500°F. Further, it has been shown (Technical Report PWA-2206, Table 144) that calcium titanate is stable up to 400 hours at 1450°F. Since the performance of this specimen was not similar to that of other calcium titanate specimens, no conclusions may be drawn concerning low-temperature changes of calcium titanate coated specimens until further testing is conducted.

G. Iron Titanate

Iron-titanium-oxide (FCT-11) was obtained from the Continental Coatings Corporation and was determined by X-ray diffraction to be Fe_2TiO_5. The iron titanate was plasma-arc sprayed onto columbium - 1 per cent zirconium tubes. A total of four specimens were tested, two of these in the total emittance rig, and the other two in the short term endurance rig.

Total Hemispherical Emittance Testing - The first specimen had a 5-mil thick coating which was dark gray, hard, and had a surface roughness equivalent to that of 320 grit emery cloth.

As can be seen in Table 14 and Figure 18, the emittance level of this specimen was about 0.87 between 1000 and 2100°F which is a value close to that obtained previously. However, at 2200°F, the coating started to volatilize appreciably, and therefore reliable data could not be obtained during cooling. After the specimen was removed from the rig, it was found that the chamber walls and instrumentation flange were coated, but the characteristics of the coating on the specimen appeared unchanged. Although the coating volatilized at 2200°F, it was decided to endurance test a specimen at 1800°F since the coating was stable at least up to a temperature of 2000°F and since data obtained up to 2100°F were equivalent to that obtained previously.

A second specimen had a plasma-arc sprayed coating of iron titanate which was 2 mils thick, gray, hard, and had a fair coating-substrate bond. The emittance was determined over a temperature range of 800°F to 1800°F. As shown in Table 15 and Figure 19, the emittance varied from 0.87 to 0.89 over the entire temperature range. This compares favorably with the first specimen tested and is indicative of good emittance reproducibility with coatings of different thicknesses.

Short Term Endurance Testing - A third specimen, which had a 4-mil thick coating of iron-titanate, was heated to 1800°F in the short term endurance rig. During heating, the emittance varied between 0.86 and 0.87 (see Table 16 and Figure 20). After the specimen had been at 1800°F with an emittance of 0.87 for 1.6 hours, a partial blockage of the cooling water caused overheating of the chamber walls and the instrumentation flange and the test was terminated. The specimen was a darker shade of gray after testing than before, but no other changes were found.

At a later date this specimen was reinstalled in the rig and a second endurance test was run at 1800°F. The emittance decreased from 0.85 at 700°F to 0.82 at 1800°F and remained constant at 0.82 (see Table 17 and Figures 21 and 22) for the remainder of the test. Although there were no visible changes in the coating after testing, it seems probable that some unidentified change had occurred inasmuch as higher values of emittance have been demonstrated by this coating in the past.

To provide further information on the stability of iron titanate, a fourth specimen was plasma sprayed with a 3-mil thick coating on a columbium - 1 per cent zirconium substrate.

The coating was dark gray, hard, and well bonded to the substrate. The specimen was heated to 1800°F and the emittance rose to 0.88. After four hours at 1800°F, a voltage lead became detached and the test was terminated to reinstrument the specimen. The voltage lead was reattached and the specimen heated to 1800°F. As can be seen in Table 18 and Figures 23 and 24, there was a large difference in temperature readings between the thermocouple and optical pyrometer. It is evident from the calculated emittance that the thermocouples were giving erroneous readings. The emittance values based on pyrometer temperature measurements are in good agreement with previous tests.

H. Nickel Chrome Spinel

Two batches of nickel-chrome spinel were prepared by heating stoichiometric mixtures of nickel oxide (NiO) and chromic oxide (Cr_2O_3) at 2500°F for 10 hours. One batch was heated in air and the other in an oxygen-rich atmosphere. X-ray diffraction analyses of the resulting material revealed 70 per cent conversion to the spinel ($NiO \cdot Cr_2O_3$) in the first batch and 80 per cent in the second. In all, five specimens were prepared for emittance testing by plasma-arc spraying the spinel mixtures onto columbium - 1 per cent zirconium tubes.

Total Hemispherical Emittance Testing - The total hemispherical emittance of several specimens was determined to evaluate reproducibility of emittance from specimens prepared at different times, and also to compare the effect of the spinel content of the coating on emittance.

A 2-mil thick coating of the 70 per cent spinel mixture was plasma-arc sprayed onto a columbium - 1 per cent zirconium tube. As was the case with crystalline boron, difficulty was encountered in obtaining a thick coating. The coating was black, soft, and had a matte texture similar to that of 240 grit emery cloth. The coating-substrate bond strength was poor.

The total hemispherical emittance was measured between 300 and 2200°F and results appear in Table 19 and Figure 25. The emittance increased from about 0.78 at 300°F to 0.87 at 900°F. It remained at 0.87 between 900°F and 2100°F. At 2200°F, a bright spot appeared at the bottom of the tube and more power was required to maintain the temperature level. During cooling, the emittance data retraced that obtained during heating.

After the specimen was removed from the rig, it was found that the lower part of the specimen which had overheated during testing had turned white while the remainder of the specimen remained black. The black coating was fairly hard and now had a fair coating-substrate bond strength. A shiny metal coating was found on the interior of the chamber and on the instrumentation flange. It appears that there was some defect or impurity in the coating at the lower end of the specimen since this is the only place where an observable change in the coating occurred.

The second specimen was coated with the 80 per cent spinel mixture by plasma-arc spraying, but, unlike the spraying of previous specimens, 2.5 per cent hydrogen was blended with the argon plasma gas to raise the plasma gas temperature. Pure argon was used for the carrier gas. The resulting coating was 4 mils thick, dark gray, and hard. The coating - substrate bond was good, and the texture was similar to that of 240 grit emery cloth. As shown in Table 20 and Figure 26, the emittance remained at 0.90 from 800°F to 1800°F. This value is higher than previously reported and may be the result of the higher spinel percentage. No changes in the appearance of the coating were observed as a result of testing.

Short Term Endurance Testing - Three short term endurance tests were conducted to determine the emittance stability as a function of time. These tests were performed in the short term endurance rig.

The first specimen had a 4-mil thick coating of the 70 per cent spinel mixture. The coating was black, fairly hard and was well bonded to the substrate. Testing was conducted at 1800°F and results appear in Table 21 and Figure 27. The emittance values were much lower than previously reported for this material. Further, after less than 10 hours of exposure to a temperature of 1800°F, the emittance dropped from 0.72 to less than 0.68 (see Figure 28). During cooling, the emittance remained below the values obtained during heating and the test was terminated. At the present time, no explanation can be offered for the unusually low values of emittance.

A second specimen was prepared from the 80 per cent spinel mixture. The coating was dark gray, hard, and had a good coating-substrate bond. This specimen was exposed to 1600°F for over 260 hours and, as shown in Table 22 and Figure 29, exhibited excellent stability with the emittance remaining at 0.88 over the entire exposure period.

A final specimen was prepared in the same manner as the second specimen. The coating was black, hard, and well bonded to the substrate. This endurance test was conducted at 1800°F and, as shown in Table 23 and Figure 30, the emittance decreased from an initial value of 0.88 to 0.83 after approximately 100 hours of exposure. There was some evidence of thermocouple poisoning and the reported values are therefore based on optical pyrometer readings.

It seems apparent from this series of emittance tests that nickel-chrome spinel offers good repeatability of emittance values and has good endurance capabilities up to 1600°F. At 1800°F, there is some evidence of deterioration in emittance properties and further investigation is required to establish the upper useful operating temperature of this material.

I. Silicon Carbide

Two columbium - 1 per cent zirconium tubes were coated with silicon carbide by an aluminum-phosphate bonding procedure described in Section IV. The first specimen was bluish white, moderately hard, and fairly well bonded to the substrate. The emittance values were somewhat erratic with the highest readings being recorded at 300°F (see Table 24 and Figure 31) followed by generally decreasing values up to 1300°F at which temperature the coating failed. Some flakes of the separated coating were measured and found to be 8 mils thick. It appears probable that this excessive thickness contributed to the coating failure. These values of emittance are somewhat lower than expected for silicon carbide. It is uncertain whether this is a result of the surface finish or should be attributed to the aluminum phosphate binder. The second specimen coating was gray, moderately hard, and not well bonded to the substrate. The emittance values were again erratic, but at a somewhat higher level than those of the first specimen (see Table 25 and Figure 32). Emittance measurements were made over a temperature range of 300°F to 1400°F. Upon further heating to 1500°F, the coating failed and the test was terminated. It is evident from these tests that better bonding techniques must be developed before silicon carbide can be considered as a high emittance coating for high temperature applications.

J. Spinel Enamel

Two AISI-310 stainless steel tubes were coated with a high temperature enamel coating by the A. O. Smith Corporation. The coating contained a large percentage of iron spinel and was fired at 1600°F.

Prior to testing, the coatings were hard, glossy black, and well bonded to the substrates. The coatings ranged from 3 to 4 mils in thickness.

The design of the specimen holder requires that the tube ends be flattened in order to fit the end grips. During the flattening of the first tube, the coating fractured and separated from the substrate in several places, making the specimen unsuitable for testing. This problem was alleviated in the second specimen by preheating the tube ends before flattening.

This specimen, was mounted in the total hemispherical emittance rig and the emittance was determined over a temperature range of 800°F to 1700°F.

As shown in Table 26 and Figure 33, the emittance increased from 0.84 at 800°F to 0.89 at 1600°F. The temperature was then lowered to 1400°F and the emittance values essentially repeated the previous readings. The temperature was then increased to 1700°F and the emittance again rose to 0.89. At this temperature, the coating separated from the tube and the test was terminated.

III COATING ENDURANCE TESTS IN SUPPORT OF NASA SPACE POWER SYSTEMS

A. Introduction

The long term endurance testing of four finned-tube radiator segments has been completed. Thermocouple data indicated a relatively constant temperature profile across each of the specimens throughout the tests and therefore showed no significant emittance changes for any of the specimens. During the latter part of the tests the profile data was similar to that reported in Technical Report PWA-2206.

Post-test analyses were conducted for each specimen and test chamber. The specimens were cooled to room temperature in vacuum and then the residual gas in the chambers was analyzed using a Veeco Model GA-3 residual gas analyzer connected to the system by the method shown in Figure 34. Contamination of the residual gas was avoided by evacuating and baking out the system external to the chamber to a pressure below that in the chamber before opening the connecting valve. The residual gas analyses indicated that the increase in noble gases (which are not removed by ion-gettering pumps) was not great enought to significantly distort pressure readings made by monitoring the ion-gettering pump current.

Following analysis of the residual gases, the pumps were turned off and the leak rate of each chamber was determined. Leak rates were determined to indicate the rate of air-flow into the test chambers to facilitate analysis of chemical changes in the specimens in the event that such changes had occurred. The leak rates were all found to be well below the rates at which a detrimental amount of flow would occur. The chambers were then re-evacuated with pressure measurements being made using both the ion pump current and the ionization gage in the external system to check the accuracy of the vacuum measurements made during endurance testing.

The chambers were then vented with dry nitrogen and opened. The finned-tube segments were removed, cut apart, and subjected to chemical and metallurgical analyses.

Included in the analyses was the measurement of coating bond strength. This was determined by bonding a portion of the coated fin between the two end grips shown in Figure 35 with epoxy resin. The epoxy resin joints were cured at 350°F for one hour to produce a bond of 12,000 psi which is in excess of the coating-substrate bond strength. The assembly was then subjected to tensile testing. Failure could occur at three locations. If it occurred within the epoxy, it would indicate that the coating-to-substrate bond strength was greater than 12,000 psi. If it occurred within the coating, then the interparticle bond strength was lower than that between the coating and the substrate. Failure at the coating-substrate interface would indicate a coating-to-substrate bond strength which was lower than the interparticle strength.

B. Endurance Test Number 1, SNAP-8 Test Section

This test section was coated with an aluminum-phosphate bonded mixture of nickel-chrome spinel ($NiO \cdot Cr_2O_3$) and silicon dioxide and completed 15,000 hours of endurance testing at a fin root temperature of 700°F in a vacuum of about 10^{-7} mm Hg. After 2700 hours of testing, the specimen was inadvertently overheated causing the coating on the tube portion of the specimen to wrinkle and the thermocouples near the fin root to become poisoned. Testing was continued until a total of 7200 hours had been accumulated at which time the specimen was reinstrumented. The test was subsequently continued to accumulate a total running time of 15,000 hours. No additional changes in the coating occurred during testing. The segment is shown after testing in Figures 36 and 37.

The results of the residual gas analysis are shown in Table 27, and indicate that the concentration of argon increased slightly while that of the other gases decreased slightly. This behavior is a result of the selective pumping characteristics of ion gettering pumps. The results of the leak test are shown in Figure 38.

Metallurgical and chemical testing was conducted on samples taken from the locations shown in Figures 39 and 40. Spectrographic and X-ray analyses (Table 28) detected no significant changes in the chemical composition or crystalline structure of the sample as a result of testing although a small amount of silicon carbide, be-lived to be an impurity present in the original coating, was detected. Micro-hardness testing (Table 29) indicated that the hardness of the segment was typical of the aluminum alloys of which the specimen was made (1100 alloy for the fin and 6061 alloy for the tube).

Photomicrographs of the fin and wrinkled portion of the tube were taken. The structure of the fin (Figure 41) was typical of 1100 aluminum alloy. Photomicrographs of the wrinkled area (Figure 42), revealed the build-up of a brittle material which is believed to be silicon at the base of the wrinkles. The grain size in the vicinity of the wrinkles was found to be considerably larger than that in unaffected portions of the tube. Although metallurgical changes have occurred in this region, the cause of wrinkling is uncertain because the effect on the structure of 12,000 additional hours of endurance testing after overheating cannot be adequately evaluated.

The results of mechanical testing are shown in Table 30. Although the coating had the lowest bond strength of those subjected to long term endurance testing, it also withstood the greatest bend angle of any of the coatings tested. The coating-to-substrate bond strength was greater than the interparticle bond strength.

The thermocouples were checked and it was found that no poisoning had occurred after replacement of the original thermocouples at 7200 hours.

Post-test analyses of the nickel-chrome-spinel-and-silicon-dioxide coated segment have indicated that there was no interaction between the aluminum substrate and the coating and that, aside from the effects of overheating at 2700 hours, there was no change in the coating as a result of testing.

C. Endurance Test Number 2, SNAP-8 Test Section

This SNAP-8 radiator segment was plasma-arc sprayed with a coating of "Titania Base" powder obtained from the Plasmadyne Corporation. The powder is primarily titanium dioxide but contains small amounts of other oxides. The specimen was endurance tested for 14,037 hours at a fin root temperature of 700°F in a vacuum of about 10^{-7} mm Hg. Cracks in the coating on the tube portion of the specimen were first observed at 2810 hours (Figure 43) and spalling was observed at 6840 hours (Figure 44). The coating continued to deteriorate during the remainder of the test. After approximately 12,287 hours, the vacion pump stalled and the power to the specimen was shut off. The pump was subsequently restarted and the test continued, but the thermal cycling to which the specimen was subjected caused additional coating loss. The test was concluded after a total endurance time of 14,037 hours. Figure 45 shows the appearance of the specimen at the end of the test and Figures 46 and 47 show the specimen after the bell jar was removed. As shown, removal of the bell jar caused a small additional amount of coating to fall off the tube.

The results of the residual gas analysis (Table 31) showed an increase in the concentration of argon and a decrease in the concentration of the other gases as a result of the selective pumping of the ion-gettering pump. The results of the leak test appear in Figure 48.

Figures 39 and 49 show the locations from which the various test samples for chemical and metallurgical analyses were taken. The results of spectrographic and X-ray analyses (Table 28) indicated that no chemical or crystalline structural changes occurred as a result of testing, and microhardness tests (Table 29) indicated that the material hardness was typical for the aluminum alloys of which the segment was made.

Photomicrographs (Figure 50) showed that the structure of the specimen was typical of the alloy and revealed no evidence of interdiffusion between the coating and the aluminum substrate.

Mechanical testing (Table 30) indicated that the coating was quite brittle, but that it was well bonded to the fin. The interparticle bond strength was lower than the coating-to-substrate bond strength.

The thermocouples were checked and there was no evidence of significant thermocouple poisoning.

Post-test analysis has, therefore, indicated that although a certain amount of spalling from the tube portion of the specimen occurred during testing, the remainder of the coating remained well bonded to the substrate and was unchanged as a result of testing.

D. Endurance Test Number 3, Sunflower I Test Section

The Sunflower I test section was coated with the same type of "Titania Base" powder used for the SNAP-8 section described in the preceding section. Endurance testing was conducted at a fin root temperature of 650°F in a vacuum of about 10^{-7} mm Hg. After 12,691 hours, a line voltage fluctuation caused the electromagnetic relay to shut off the specimen power. The specimen was maintained in vacuum and examined before being returned to the endurance temperature. No change in the coating resulted from thermal cycling and the test was continued until 13,755 hours were accumulated and the relay again shut off power to the specimen. At this time testing was permanently discontinued. As shown in Figures 51, 52, and 53, no change in the appearance of the coating occurred as a result of testing.

The results of the residual gas analysis (Table 32) showed that the concentration of argon increased as a result of the selective pumping of the ion gettering pump. There was also an increase in the concentration of some hydro-carbons, but the reason for the increase is not known. The results of the leak test are shown in Figure 54 and indicate that this rig had the lowest leak rate of the four long term endurance rigs.

The locations of the various samples for metallurgical and chemical analysis are shown in Figures 55 and 56. X-ray and spectrographic analyses (Table 28) indicated that no change in the chemical composition or crystalline structure of the coating occurred as a result of testing. Microhardness tests (Table 29) indicated that the hardness of the samples was typical for the material in the annealed condition. Photomicrographs (Figure 57) show normal structures for the material and indicate that no interaction between the coating and substrate occurred. Mechanical testing (Table 30) indicated that the properties of this coating were similar to those of the titania base coating tested on a SNAP-8 test section. The thermocouple calibration check indicated that the thermocouples were stable throughout the test.

Post test analysis of the titania base coated Sunflower I test section has indicated that the coating was unchanged by the endurance testing and had properties similar to those of the titania base coated SNAP-8 test section. It is noted, however, that no spalling occurred on the tube portion of the Sunflower I test section whereas extensive spalling occurred on the tube portion of the SNAP-8 test section. This is attributed to the use of type 347 stainless steel for the tube portion of the Sunflower I test section which provided a tube with a lower coefficient of thermal expansion than the aluminum 6061 alloy used for the SNAP-8 section tube.

E. Endurance Test Number 4, SNAP-8 Test Section

A mixture of silicon carbide and silicon dioxide was aluminum-phosphate bonded to a SNAP-8 test section and endurance tested for 12,781 hours at a fin-root temperature of 700°F in a vacuum of about 10^{-7} mm Hg. No change in the appearance of the test section occurred as a result of testing. Figures 58 and 59 show the section after testing.

The residual gas analysis (Table 33) of the test chamber indicated that the concentration of argon increased and that the concentration of the other gases either decreased or remained essentially unchanged. Results of the leak test appear in Figure 60.

Test samples for metallurgical and chemical analysis were taken from the locations shown in Figures 39 and 61. Spectrographic and X-ray analysis (Table 28) indicated that no significant changes in the coating occurred as a result of testing. Microhardness tests (Table 29) indicated hardnesses typical of aluminum alloys. Photomicrographs (Figure 62) show normal structures and indicate that interactions between the coating and the substrate did not occur. Mechanical testing (Table 30) indicated that this coating was ductile, but had a low coating-to-substrate bond strength. The thermocouple calibration check indicated that no appreciable thermocouple poisoning occurred during testing.

F. Summary

Long term endurance testing four coated space radiator segments has demonstrated the overall stability of coatings of aluminum-phosphate bonded silicon carbide, aluminum-phosphate bonded nickel-chrome spinel, and plasma-arc sprayed titania base when bonded to aluminum fins and exposed to temperatures of about 700°F in a vacuum of about 10^{-7} mm Hg. None of the coatings reacted with the fins and no changes in either the coating or fin material resulted from endurance testing. The coatings remained satisfactorily bonded to the fins with coating-to-substrate bond strengths which were higher than the inter-particle bond strengths. The aluminum-phosphate bonded coatings were weaker, but more ductile than the plasma-arc sprayed titania base coatings. Coating failures occurred only on the tube portion of two specimens and in one case could be directly attributed to overheating the specimen.

The tests also demonstrated the feasibility of endurance testing in vacuum for periods extending up to 15,000 hours. Although the concentration of argon increased slightly, the ion-gettering pumps operated satisfactorily in all cases, and, with the exception of difficulties caused by overheating during the testing of the nickel-chrome spinel coated SNAP-8 radiator segment, the instrumentation remained stable throughout the tests.

IV. INVESTIGATION OF ALKAPHOS-BONDED COATING PROCEDURES

Silicon carbide has been found to have an emittance of better than 0.90, but difficulty has been encountered in maintaining an adequate bond between silicon carbide and a substrate at temperatures above 1400°F. To date, only thermal spraying and aluminum phosphate bonding techniques have been found to be suitable for high temperature applications. However, silicon carbide decomposes when applied by thermal spraying, and therefore efforts have been directed toward developing a satisfactory technique for bonding silicon carbide with aluminum phosphate.

A stable aluminum phosphate compound, anhydrous aluminum metaphosphate ($Al_2O_3 \cdot 3P_2O_5$), is formed at 930°F[3] with the loss of the last of the chemically combined water. It would be expected, therefore, that, in the absence of thermal shocking, an aluminum-phosphate bonded coating which remained bonded up to 930°F would remain bonded up to at least 2000°F. Aluminum phosphate bonded coatings of silicon carbide, however, have separated at temperatures around 1400°F. To determine the cause of this behavior, an investigation of substrate surface preparation and curing procedures is being conducted.

Since the source of aluminum-phosphate solution used for bonding has not been found to influence emittance data, the investigation has been confined to one commercially available solution, namely, Alkaphos C, a product of Monsanto Chemical Company. This product is particularly stable which enables a single batch of material to be used throughout the investigations and therefore precludes the possibility of variations in mixing practice influencing the test results. AISI-310 stainless steel strips and columbium-1 per cent zirconium tubes have been used for substrates.

To determine the effects of surface roughness, four columbium-1 per cent zirconium tubes were prepared with varying degrees of roughness. The processes used were chemical cleaning, vapor blasting, grit blasting with 90 mesh alumina, and grit blasting with 28 mesh steel. Each tube was cleaned with trichloroethylene, flushed with water, and rinsed with acetone. The coating and curing procedure used is outlined in

[3]Eubanks, A. G. and Moore, D. G., "Investigation of Aluminum Phosphate Coatings for Thermal Insulation of Airframes," NASA TN D-106, National Aeronautics & Space Administration, Washington, D.C., November, 1959.

Table 34, specimens numbers 1 through 4. Although some crumbling occurred on all of the samples, the coating applied to the tube which was grit blasted with 90 mesh alumina showed the least tendency to separate from its substrate.

Various curing cycles were investigated for Alkaphos-bonded silicon-carbide coatings on AISI-310 stainless steel strips and on columbium-1 per cent zirconium tubes. The substrates were prepared by either vapor or grit blasting and were degreased and rinsed with acetone immediately before being coated. Coatings of various compositions (see Table 34) were applied by spraying and were dried and cured for various times and at various temperatures. All of the specimens which were heated above 400°F were furnace-cooled to 400°F to prevent thermal shocking. Results of these tests appear in Table 34, specimens numbers 5 through 14. Coatings which were cured at temperatures above 400°F remained intact until after the specimens were removed from the furnace, but after a few hours the coatings crumbled. Since this problem had not been encountered previously when a more acidic aluminum-phosphate solution made by Pratt & Whitney Aircraft had been used, it was thought that increasing the acidity of the Alkaphos might reduce the amount of crumbling. A specimen was prepared using a slurry to which phosphoric acid had been added, but excessive spalling of the coating resulted (specimen number 15 in Table 34). To gain further insight into the problem, a coating with $SrO \cdot TiO_2$ filler was applied and cured at a temperature above 400°F. No crumbling occurred (Table 34, specimen number 16) which indicates that the process is suitable for some materials.

As may be seen in Table 34, all of the silicon carbide coatings bubbled during curing. In an attempt to determine the cause of the bubbling, coatings of Alkaphos C without filler material were applied to a stainless steel and to a columbium-1 per cent zirconium substrate. Curing included heating to 950°F. No bubbling occurred, but the coatings were still tacky at completion of the curing cycle indicating incomplete curing (see specimens 17 and 18 in Table 34). The lack of bubbling, however indicates that the Alkaphos C does not, in itself, cause the bubbling. It was noted that the higher-purity green silicon carbide was not so prone to bubbling as was the black silicon carbide. On this basis it is considered possible that the bubbling is caused by impurities which react with the aluminum phosphate solution.

To further evaluate the cause of bubbling, a coating containing $CaO \cdot TiO_2$ filler material was applied to a columbium-1 per cent zirconium tube. This specimen (specimen number 19 in Table 34) was cured by the process which had been most successful with silicon carbide coatings. No bubbling occurred.

On the basis of work completed to date and consultation with the Monsanto Chemical Company, it appears that silicon carbide is a particularly difficult material to bond by aluminum phosphate solutions. The best coatings produced to date were air-dried for 20 hours, and oven cured at 200°F for 2 hours, 250°F for 2 hours, 300°F for 2 hours, and 400°F for 2 hours.

To further investigate Alkaphos-bonded coating procedures, a coating containing green colored silicon carbide and cured by the method described above was tested in the total hemispherical emittance rig. The coating was 6 mils thick, contained a few small bubbles, and was still tacky at the end of the curing cycle, indicating incomplete curing. The appearance of the coating before testing is shown in Figure 63. Data was not taken below 1000°F since previous silicon carbide coatings had been stable up to 1300°F. However, the data taken at 1000°F for the present coating indicated that the coating was already failing. The appearance of the coating after testing is shown in Figure 64.

In an attempt to produce better coatings, additional columbium - 1 per cent zirconium tubes were coated using a modified procedure. A slurry was formed by mixing 100 grams of silicon carbide with 100 milliliters of Alkaphos C and then adding a mixture of 0.1 gram of Trixton X100 (a polyethylene glycol nonyl phenol procured from Rohm and Haas Company) and 5 grams of distilled water. The slurry was thoroughly mixed, subjected to a reduced pressure of 27 inches of Hg until no additional air bubbles appeared, and then let stand overnight. The tubes to be coated were placed on a horizontal spindle, washed with acetone, and dried. They were then placed 12 inches above a hot plate set at 700°F and rotated at 20 RPM while thin coats of slurry were applied with a brush. The specimens were rotated after coating until they achieved a dull luster, were then removed from the spindle, mounted in a frame so that only the tube ends touched the frame, and placed in a forced-air oven. The oven temperature was raised to 250°F over a two-hour period and then held at 250°F for a minimum of 4 hours. The specimens were then moved to a vacuum oven preheated to 250°F and the pressure was reduced to 27 inches of Hg over a one-half hour period. The oven temperature was increased to 400°F at the rate of 33°F per hour and conditions were then maintained for 16 hours. At the end of this period the specimens were slowly cooled under reduced pressure. The resulting coatings were uniform and contained no bubbles.

The behavior of these coatings under test conditions is described in the section on silicon carbide coatings. The appearance of the coatings before and after testing is shown in Figures 63 and 64 respectively.

V. INVESTIGATION OF TOTAL HEMISPHERICAL EMITTANCE RIG DISCREPANCIES

As reported previously in Pratt & Whitney Aircraft Technical Report PWA-2163, difficulty had been encountered in reconditioning the total hemispherical emittance rig after a coating of manganese oxide volatilized and coated the instrumentation flange and other parts in the chamber with a metallic coating. A complete cleaning of the interior of the chamber and replacement of the affected electrical components failed to re-establish the previous performance. Since total hemispherical emittance measurements depend on measurements of specimen power input and specimen surface temperature, the components involved with these measurements were carefully analyzed. Power is measured in this rig with a precision AC-DC voltmeter in conjunction with several current shunts. The voltmeter used was checked and found to be accurate to within 0.2 per cent and the current shunts were checked and found to deviate from their nominal values by not more than 0.08 per cent.

Comparison of values obtained with platinum-platinum 10 per cent rhodium thermocouples (which are used extensively in the total hemispherical emittance rig) with values obtained with chromel-alumel thermocouples revealed a consistent discrepancy, with the platinum-platinum 10 per cent rhodium thermocouples yielding lower values. Initial investigation of the thermocouples evaluated the effect of the substrate material on thermocouple operation. Both platinum-platinum 10 per cent rhodium and chromel-alumel thermocouples were attached to uncoated tubes of columbium-1 per cent zirconium, AISI-310 stainless steel and tantalum. It was found that the temperature measurement discrepancy was the same with all substrates and it was therefore concluded that the substrate material was not the cause of the discrepancy.

Analysis was then directed to the thermocouple wire itself, and it was discovered that one roll had not been properly annealed. This roll had been started at the time of the volatilization of the manganese oxide in the total hemispherical emittance rig. To confirm that the improper preparation of the wire was the cause of the discrepancy, temperature measurements were made using 3-mil diameter chromel-alumel thermocouples and both 1-mil and 3-mil diameter platinum-platinum 10 per cent rhodium thermocouples which were properly annealed. Agreement between the 3-mil diameter platinum-platinum 10 per cent rhodium and the chromel-alumel thermocouples was within a few degrees in the range of 200 to 1800°F (see Table 35). As shown in Table 36, agreement was also good between the 1- and 3-mil diameter platinum-platinum 10 per cent rhodium thermocouples, thus confirming the theoretical analysis of thermocouple lead heat conduction losses presented in Appendix L of Pratt & Whitney Aircraft Technical Report PWA-2206. In conjunction with the thermocouple investigations, emittance data was obtained for AISI-310 stainless steel and tantalum and are reported in Sections II-A and II-B of this report.

The results of this investigation have resolved the difficulties with the total hemispherical emittance rig and the rig has been returned to service.

VI EMITTANCE MEASUREMENTS OBTAINED IN 1962 CORRECTED FOR TOTAL HEMSIPHERICAL EMITTANCE RIG DISCREPANCIES

During the period at the end of 1962 when the accuracy of data obtained with the total hemispherical emittance rig was under investigation, emittance data was obtained for a number of materials, but was withheld pending resolution of the problems associated with temperature measurement. Since these problems have now been resolved, it is possible to report these data:

A. Hastelloy C (Partially Oxidized)

Partially oxidized Hastelloy C powder was obtained from the Haynes Stellite Company for emittance testing. The material was plasma-arc sprayed onto an AISI-310 stainless steel tube to produce a 7-mil thick coating which was metallic gray, very hard, and well bonded to the substrate. The coating had a texture similar to that of 40 grit emery cloth. Emittance measurements were made between 300°F and 1700°F and the results (Table 37 and Figure 65) show that the emittance increased slowly from about 0.54 at 300°F to about 0.62 at 1700°F. During cooling the emittance duplicated the values obtained during heating. Testing was not attempted above 1700°F since the emittance was low and since there was danger that the nickel in the alloy might volatilize at higher temperatures. Had the powder been completely oxidized, the emittance values probably would have been higher[4] and the nickel would have been combined with an oxide and not so liable to volatilize. No change in the appearance of the coating resulted from testing.

B. Hastelloy X (Partially Oxidized)

Hastelloy X powder was obtained from the Haynes Stellite Company with particle diameters between 44 and 105 microns. An 8-mil thick coating of the material was applied to an AISI-310 stainless steel tube by plasma-arc spraying. The resulting coating was gray with silvery metallic specks, hard, and well bonded to the substrate. The texture was similar to 40 grit emery cloth. Emittance testing was confined to temperatures between 300 and 1700°F for the reasons discussed in the preceding section. Results are presented

[4] Wade and Casey, op. cit.

in Table 38 and Figure 66 and show that the emittance increased from 0.59 at 300°F to about 0.66 at 1700°F and then repeated the same values during cooling. After testing it was found that the silver specks had darkened to a golden color, but no other changes were apparent.

C. Oxidized Kennametal K-151A

Kennametal K-151A powder was oxidized by the supplier, Kennametal, Incorporated, for 20 minutes at 1600°F so that the emittance data obtained could be compared with that of oxidized sheets of the same material tested by Wade and Casey.[5] A 4-mil thick coating of the material was applied to an AISI-310 stainless steel tube. The resulting coating was dark gray, fairly hard, and had a texture similar to that of 40 grit emery cloth. The coating-to-substrate bond was fair.

The specimen was tested between 500°F and 1700°F and results appear in Table 39 and Figure 67. The emittance increased from 0.75 at 500°F to about 0.90 at 1300°F and then remained constant up to 1700°F. Two emittance points were obtained during cooling and these indicated that no changes in the coating occurred during the test. When the specimen was removed from the rig, its appearance was found to be unchanged. Agreement between the data obtained by Pratt & Whitney Aircraft and that obtained by Wade and Casey is good when it is recognized that the Pratt & Whitney Aircraft data was obtained in vacuum using a plasma-arc sprayed coating whereas Wade and Casey measured total normal emittance in air using a flat sheet of Kennametal K-151A.

D. Oxidized Kennametal K-162B

Similar to the Kennametal K-151A powder, Kennametal K-162B powder was oxidized by the supplier for 20 minutes at 1600°F. A 5-mil thick coating was produced by plasma-arc spraying on an AISI 310 stainless steel tube. The coating was dark gray, fairly hard and had a texture similar to that of 40 grit emery cloth. The coating-substrate bond strength was fair.

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[5] Wade and Casey, op. cit.

Emittance measurements were made between 300 and 1700°F and the results are presented in Table 40 and Figure 68. As shown, the emittance increased linearly from 0.77 at 300°F to 0.89 at 1700°F and then repeated the same values during cooling. No change in the coating was apparent after testing.

Although at temperatures above 800°F, the emittance values obtained were slightly higher than those obtained by Wade and Casey, the difference is presumably attributable to the different procedures used and in general the agreement is reasonable.

E. Barium Titanate

Barium titanate powder ($BaO \cdot TiO_2$) was obtained from the Continental Coatings Corporation under the material designation FCE-11 and a 7-mil thick coating of the material was aluminum phosphate bonded to a columbium - 1 per cent zirconium tube. The coating was white, soft, and poorly bonded to the substrate. The texture was similar to that of 320 grit emery cloth.

Emittance measurements were made between 300°F and 1000°F and the data are presented in Table 41 and Figure 69. The emittance varied from 0.85 at 300°F to 0.63 at 1000°F and was lower during cooling than during heating, indicating that a change in the coating had occurred. After testing, the color of the coating was irregular although still white. A small amount of powder spalled from the specimen during testing and was found on the bottom heat shield.

F. Calcium Titanate

Two columbium - 1 per cent zirconium tubes were plasma-arc sprayed with a high purity calcium titanate ($CaO \cdot TiO_2$) powder obtained from the Titanium Division of the National Lead Company.

The first specimen had a 5-mil thick coating containing white crystals on a blue background. The coating was hard, well bonded to the substrate, and had a texture similar to that of 40 grit sand-paper. Total hemispherical emittance data was obtained between 300°F and 1600°F. As shown in Table 42 and Figure 70, the emittance decreased during run 1 from 0.75 at 300°F to 0.71 at 900°F and then increased until a value of about 0.94 was attained at 1500°F. During cooling the emittance decreased to 0.86 at 800°F. The specimen was then reheated with similar emittance values being obtained. After testing, the white crystals in the coating were still present, but the background had turned gray. No other changes were apparent.

The second specimen had a 4-mil thick gray coating which was hard and well bonded to the substrate. The texture was similar to that of 40 grit emery cloth. Emittance values were obtained between 300 and 1500°F and are presented in Table 43 and Figure 71. Comparison of Figures 70 and 71 reveals that the general shapes of the curves are similar, but that the emittance of the second specimen was slightly higher. No changes in the appearance of the specimen occurred as a result of testing.

G. Iron Titanate

A 3.5-mil thick coating of iron titanate was aluminum phosphate bonded to a columbium - 1 per cent zirconium tube. The coating had a texture similar to that of 320 grit emery cloth, was well bonded to the substrate, and was light brown except around the black-body holes where the color was somewhat darker.

Emittance measurements were obtained between 300 and 1900°F and the results are presented in Table 44 and Figure 72. As shown in Figure 72, the curve is S shaped with maximum values of 0.87 and 0.83 being obtained at 500 and 1500°F respectively. Between 1600 and 1700°F one of the thermocouples failed and at 2000°F the voltage leads failed, terminating the test. After testing the specimen was black rather than the original light brown. Data obtained during this test using optical pyrometer temperatures confirm previous data for iron titanate coatings (see Pratt & Whitney Aircraft Report PWA-2206).

H. Iron Titanate With Alumina

Iron Titanate with alumina was aluminum phosphate bonded to a columbium - 1 per cent zirconium tube. The resulting coating was 4 mils thick, light brown, and hard. The coating was well bonded to the substrate and had a texture similar to that of 320 grit emery cloth.

Emittance data was obtained between 300 and 2000°F and are presented in Table 45 and Figure 73. The emittance decreased from 0.83 at 500°F to a low of 0.70 at 1300°F, and then increased to 0.93 at 2000°F. During cooling, the emittance decreased, but remained higher than during initial heating. At 1400 and 1500°F, the temperature profile along the specimen was not even although the specimen had an even glow along its length. Shining a light on the specimen, however, revealed that the coating had a spotty appearance at 1600°F which was not observed at either 1500 or 1700°F. Further, a large increase in power was required to attain and maintain the specimen temperature at 1700°F, indicating that a change in the coating occurred at this temperature resulting in the higher subsequent emittance values. After testing, the coating was black, brittle, and poorly bonded to the substrate. The texture remained unchanged.

I. Strontium Titanate

Three strontium titanate coated specimens were tested. One of these was obtained by aluminum-phosphate bonding a commercial grade of strontium titanate obtained from the Plasmadyne Corporation to an AISI-310 stainless steel tube. The other two coatings were obtained by plasma-arc spraying a high purity grade of strontium titanate obtained from the Titanium Division of the National Lead Company to columbium -1 per cent zirconium substrates.

Aluminum Phosphate Bonded Coating - The aluminum phosphate bonded coating was 10 mils thick, tan, and soft. The coating-substrate bond strength was fair and the coating had the texture of 40 grit emery cloth.

Emittance data was obtained between 300 and 1500°F and are presented in Table 46 and Figure 74. As shown, the emittance decreased from 0.65 at 300°F to 0.38 at 1500°F. The points obtained during cooling indicated that a permanent change in the coating had occurred. As the specimen was cooled below 1050°F, the coating shattered and violently separated from the substrate. Comparison of emittance curves for barium, calcium, and strontium titanates (Figures 69, 70, and 74 respectively) indicates that the decrease in emittance with increasing temperature is characteristic of titanates.

Plasma-Arc Sprayed Coatings - The first plasma-arc sprayed coating was 3 mils thick and contained white crystals on a blue background. The coating was hard, well bonded to the substrate, and had a texture equivalent to that of 80 grit emery cloth. Emittance data was obtained between 300 and 1600°F and is presented in Table 47 and Figure 75. During the first run, the specimen was heated to 1500°F and the shape of the emittance curve was similar to that obtained previously with this material although the overall level was somewhat higher than previously. During heating, the emittance rose from 0.83 at 300°F to 0.93 at 1400°F. During cooling, the emittance remained at a level higher than during heating and the higher values were repeated during subsequent thermal cycling, indicating that the change was permanent. A similar phenomenon has been previously observed with a calcium titanate coating although heating the calcium titanate coating to 1700°F resulted in a deterioration in emittance properties. After testing, the coating color was blue-gray.

The second plasma-arc sprayed strontium titanate coating was 5 mils thick, hard and well bonded to the substrate. This coating contained blue crystals on a white background and had a texture similar to that of 40 grit emery cloth. Emittance data, obtained between 300 and 1500°F, appear in Table 48 and Figure 76. The emittance curve for this coating has the same general shape as that of the preceding specimen, although initially the emittance level was slightly higher. During subsequent cooling and thermal cycling, the emittance remained at a higher level than during initial heating which has been found to be typical behavior for titanate coatings. After testing the coating was gray.

J. Silicon Carbide

A 5-mil thick coating of silicon carbide was aluminum phosphate bonded to a columbium - 1 per cent zirconium tube. The coating was fairly hard and gray. The texture was coarse and the coating substrate bond was fair.

Emittance measurements were obtained between 300 and 1400°F (Table 49 and Figure 77) and varied from 0.85 at 300°F to 0.91 at 1400°F. At this temperature, dark areas were visible on the coating, indicating that a change was occurring. When the temperature was raised to 1500°F, the thermocouples separated from the substrate and the test was terminated. Examination of the specimen after testing revealed that the coating had cracked and separated from the substrate.

APPENDIX A

Tables

TABLE 1

Volatilization Test Results for Kennametal K-151A

Temperature (°F)	Pressure (mm Hg)	Remarks
1000	1.9×10^{-6}	
1200	2.8×10^{-6}	
1300	2.5×10^{-6}	Coating attained uniform brightness
1400	2.9×10^{-6}	
1500	3.6×10^{-6}	
1600	4.0×10^{-6}	
1700	8.1×10^{-6}	
1750	9.8×10^{-6}	Dark spots appeared on coating
1800	1.4×10^{-5}	
1850	2.3×10^{-5}	
1900	3.4×10^{-5}	
1950	5.6×10^{-5}	Noticeable plating on glass bell jar but no shadows visible
2000	8.6×10^{-5}	
2050	1.2×10^{-4}	
2100	1.5×10^{-4}	
2150	1.9×10^{-4}	
2200	2.7×10^{-4}	
2250	1.9×10^{-4}	
2300	1.5×10^{-4}	
2350	1.1×10^{-4}	Large power increase required to attain temperature
2400	1.2×10^{-4}	
2450	1.4×10^{-4}	
2500	7.6×10^{-5}	Small power increase required to attain temperature
2600	7.6×10^{-5}	Shadows visible on glass bell jar; specimen temperature drifted downward at constant power setting

TABLE 2

Volatilization Test Results for Kennametal K-162B

Temperature (°F)	Pressure (mm Hg)	Remarks
1000	2.2×10^{-6}	
1200	3.1×10^{-6}	
1300	2.8×10^{-6}	
1400	3.3×10^{-6}	
1500	3.9×10^{-6}	
1600	4.1×10^{-6}	
1700	6.0×10^{-6}	Specimen temperature drifted downward at constant power setting; dark spots appeared on coating
1750	8.0×10^{-6}	Specimen temperature slowly drifted downward at constant power setting; no further change in appearance of coating
1800	1.3×10^{-5}	
1850	2.5×10^{-5}	
1900	4.0×10^{-5}	Stainless steel square became milky in appearance
1950	4.7×10^{-5}	
2000	6.6×10^{-5}	
2050	8.2×10^{-5}	
2100	1.3×10^{-4}	
2150	1.4×10^{-1}	
2200	1.4×10^{-4}	
2250	1.5×10^{-4}	
2300	1.9×10^{-4}	Dark spots on coating started to disappear
2350	5.0×10^{-5}	Dark spots on coating no longer visible
2400	4.8×10^{-5}	
2450	4.8×10^{-5}	
2500	4.7×10^{-5}	
2550	4.8×10^{-5}	
2600	5.1×10^{-5}	
2611	4.9×10^{-5}	

Coating color changed from black to brown, some plating was observed when the chamber was opened.

TABLE 3

Volatilization Test Results for Iron Titanate

Temperature (°F)	Pressure (mm Hg)	Remarks
1000	3.0×10^{-6}	
1200	3.0×10^{-6}	
1400	3.6×10^{-6}	Coating attained uniform brightness
1500	4.1×10^{-6}	
1600	4.4×10^{-6}	
1700	4.2×10^{-6}	
1800	4.3×10^{-6}	
1900	4.3×10^{-6}	
2000	4.2×10^{-6}	
2100	4.9×10^{-6}	Specimen temperature drifted downward at constant power setting
2150	5.4×10^{-6}	Large power increase required to attain temperature; specimen temperature drifted downward at constant power setting
2200	8.9×10^{-6}	Small power increase required to attain temperature; specimen temperature drifted downward at constant power setting
2250	1.4×10^{-5}	Small power increase required to attain temperature; specimen temperature drifted downward at constant power setting
2300	1.6×10^{-5}	Specimen temperature drifted downward at constant power setting; shadows visible on glass bell jar

TABLE 4

Total Hemispherical Emittance

As Received, Uncoated AISI 310 Stainless Steel

Run Number	Elapsed Time (Hrs.)	Pressure (mm Hg)	Thermocouple Temp. (°F)	Thermocouple ϵ_{th}	Optical Pyrometer Temp. (°F)	Optical Pyrometer ϵ_{th}
1	0.2	4.4×10^{-7}	1000	0.264		
	0.6	3.6×10^{-7}	1202	0.276		
	0.8	3.4×10^{-7}	1401	0.285		
	1.2	9.2×10^{-7}	1600	0.289	1622	0.277
	1.6	2.0×10^{-6}	1800	0.279	1832	0.264
	1.9	2.0×10^{-6}	2000	0.287	2039	0.269
Rig Opened; Specimen Removed and Later Reinstalled						
2	0.2	1.1×10^{-6}	999	0.211		
	0.5	1.1×10^{-6}	1333	0.246		
	0.8	1.1×10^{-6}	1500	0.258	1521	0.244

TABLE 5

Total Hemispherical Emittance

Uncoated Tantalum

Run Number	Elapsed Time (Hrs.)	Pressure (mm Hg)	Thermocouple Temp. (°F)	Thermocouple ϵ th	Optical Pyrometer Temp. (°F)	Optical Pyrometer ϵ th
			First Specimen			
1	0.4	1.0×10^{-6}	1499	0.164	1516	0.158
	0.8	1.1×10^{-6}	2201	0.200	2221	0.194
	1.8	1.1×10^{-6}	2200	0.197	2226	0.190
2	2.0	1.0×10^{-6}	904	0.139		
	2.4	1.0×10^{-6}	999	0.140		
	2.6	8.7×10^{-7}	1100	0.142		
	2.9	7.5×10^{-7}	1201	0.144		
	3.2	7.3×10^{-7}	1300	0.150		
		Heating Current Off; Vacuum Maintained				
3	3.5	9.8×10^{-7}	1000	0.139		
	3.8	9.1×10^{-7}	1200	0.145		
	3.9	1.0×10^{-6}	1400	0.154		
	4.5	1.0×10^{-6}	1600	0.165	1608	0.162
	4.8	1.0×10^{-6}	1801	0.175	1817	0.170
	5.1	1.1×10^{-6}	2000	0.183	2020	0.178
	5.3	1.4×10^{-6}	2199	0.195	2226	0.187
	5.4	1.0×10^{-6}	1503	0.147	1516	0.144
	5.5	9.3×10^{-7}	1000	0.133		
			Second Specimen			
1	0.1	4.1×10^{-7}	1000	0.167		
	0.2	5.8×10^{-7}	1200	0.169		
	0.3	1.9×10^{-6}	1400	0.180		
	0.5	6.6×10^{-7}	1600	0.207	1592	0.210
	0.6	4.9×10^{-7}	1800	0.230	1789	0.234
	1.0	4.0×10^{-7}	2000	0.256	1994	0.259
	1.1	5.1×10^{-7}	2200	0.256	2215	0.250
	1.2	4.8×10^{-7}	2150	0.237	2164	0.232
	1.3	3.4×10^{-7}	1850	0.218	1859	0.215
	1.4	3.4×10^{-7}	1550	0.203	1559	0.200
	1.5	3.0×10^{-7}	1249	0.185		

TABLE 6

Total Hemispherical Emittance

Coating: Crystalline Boron - Plasma Arc Sprayed ($<$1-mil)
Substrate: Columbium - 1% Zirconium

Run Number	Elapsed Time (Hrs.)	Pressure (mm Hg)	Thermocouple Temp. (°F)	Thermocouple ϵ th	Optical Pyrometer Temp. (°F)	Optical Pyrometer ϵ th
1	0.6	4.6×10^{-7}	300	0.651		
	0.7	4.0×10^{-7}	500	0.685		
	1.0	4.4×10^{-7}	700	0.691		
	1.2	4.3×10^{-7}	900	0.705		
	1.3	8.0×10^{-7}	1000	0.710		
	1.4	5.2×10^{-7}	1100	0.721		
	1.5	5.6×10^{-7}	1200	0.728		
	1.6	4.1×10^{-7}	1300	0.736		
	1.7	1.2×10^{-6}	1400	0.740		
	1.8	6.6×10^{-7}	1500	0.747	1505	0.740
	2.3	6.2×10^{-7}	1600	0.752	1611	0.736
	2.7	1.8×10^{-6}	1700	0.757	1711	0.742
		Heating Current Off; Vacuum Maintained				
2	3.0	5.0×10^{-7}	1500	0.727	1514	0.707
	3.1	5.0×10^{-7}	1600	0.734	1611	0.718
	3.3	4.0×10^{-7}	1700	0.738	1710	0.724
	3.7	5.0×10^{-7}	1800	0.745	1811	0.731
	3.9	1.0×10^{-6}	1900	0.742	1913	0.725
	4.5	6.8×10^{-7}	2000	0.730	2019	0.707
	4.8	1.3×10^{-6}	2200	0.699	2225	0.673
	4.9	1.0×10^{-6}	2150	0.677	2169	0.657
	5.0	5.0×10^{-7}	1850	0.622	1859	0.612
	5.2	2.8×10^{-7}	1550	0.643	1562	0.643
	5.4	1.6×10^{-7}	1250	0.621		

TABLE 7

Total Hemispherical Emittance

Coating: Oxidized Kennametal K-151A - Plasma-Arc Sprayed (4-Mil)
Substrate: AISI-310 Stainless Steel

Run Number	Elapsed Time (Hrs.)	Pressure (mm Hg)	Thermocouple Temp.(°F)	Thermocouple ϵ_{th}	Optical Pyrometer Temp.(°F)	Optical Pyrometer ϵ_{th}
1	0.1	2.2×10^{-6}	800	0.820		
	0.3	2.2×10^{-6}	900	0.827		
	0.5	2.1×10^{-6}	1000	0.838		
	0.9	1.9×10^{-6}	1100	0.841		
	1.1	2.0×10^{-6}	1200	0.855		
	1.4	2.0×10^{-6}	1300	0.865		
	1.6	2.4×10^{-6}	1400	0.859		
	2.1	3.7×10^{-6}	1500	0.862	1496	0.869
	3.0	6.8×10^{-6}	1600	0.881	1592	0.895
	3.1	3.2×10^{-6}	1550	0.886	1551	0.885
	3.3	9.0×10^{-7}	1250	0.862		
	3.4	6.4×10^{-7}	950	0.851		

TABLE 8

Total Hemispherical Emittance

Coating: Oxidized Kennametal K-162B - Plasma-Arc Sprayed (5-Mil)
Substrate: AISI-310 Stainless Steel

Run Number	Elapsed Time (Hrs.)	Pressure (mm Hg)	Thermocouple Temp. (°F)	Thermocouple ϵ th	Optical Pyrometer Temp. (°F)	Optical Pyrometer ϵ th
1	0.5	5.4×10^{-7}	832	0.822		
	0.7	5.6×10^{-7}	900	0.848		
	0.8	7.8×10^{-7}	1000	0.860		
	0.9	1.4×10^{-6}	1100	0.873		
	1.2	1.8×10^{-6}	1200	0.875		
	1.3	2.3×10^{-6}	1300	0.871		
	1.4	3.2×10^{-6}	1400	0.888		
	1.6	7.6×10^{-6}	1500	0.892	1504	0.884
	1.8	1.9×10^{-5}	1600	0.900	1611	0.880
	1.9	8.6×10^{-6}	1550	0.895	1556	0.885
	2.0	1.0×10^{-6}	1250	0.874		
	2.1	5.2×10^{-7}	950	0.857		

TABLE 9

Total Hemispherical Emittance

Coating: Calcium Titanate - Aluminum-Phosphate Bonded (4-Mil)
Substrate: Columbium - 1% Zirconium

Run Number	Elapsed Time (Hrs.)	Pressure (mm Hg)	Thermocouple Temp (°F)	ϵ_{th}
1	0.2	1.8×10^{-6}	299	0.975
	0.3	1.8×10^{-6}	500	0.933
	0.5	1.5×10^{-6}	700	0.884
	0.7	1.7×10^{-6}	900	0.799
	0.9	2.0×10^{-6}	1000	0.750
	1.1	2.2×10^{-6}	1100	0.716
	1.2	2.0×10^{-6}	1200	0.657
	1.4	1.1×10^{-6}	1300	0.596
	1.6	1.2×10^{-6}	1400	0.576
	1.8	8.8×10^{-7}	1500	0.613
	2.0	6.6×10^{-7}	1301	0.632
	2.1	6.0×10^{-7}	1000	0.681

TABLE 10

Total Hemispherical Emittance

Coating: Calcium Titanate - Plasma-Arc Sprayed (5-mil)
Substrate: Columbium - 1% Zirconium

Run Number	Elapsed Time (Hrs.)	Pressure (mm Hg)	Thermocouple Temp. (°F)	Thermocouple ϵ_{th}	Optical Pyrometer Temp. (°F)	Optical Pyrometer ϵ_{th}
1	0.8	3.7×10^{-7}	300	0.778		
	2.1	3.1×10^{-7}	500	0.814		
	2.5	3.2×10^{-7}	700	0.830		
	3.1	1.0×10^{-6}	900	0.850		
	3.6	1.3×10^{-6}	1000	0.856		
	3.8	5.0×10^{-7}	1100	0.861		
	4.1	4.7×10^{-7}	1198	0.884		
	4.4	5.7×10^{-7}	1300	0.897		
	4.7	1.4×10^{-6}	1400	0.900		
	4.8	1.2×10^{-6}	1500	0.904	1511	0.884
	5.0	7.9×10^{-7}	1252	0.880		
2	5.2	1.6×10^{-6}	1601	0.895	1608	0.883

TABLE 11

Total Hemispherical Emittance

Coating: Calcium Titanate-Plasma-Arc Sprayed (2-Mil)
Substrate: Columbium - 1% Zirconium

Run Number	Elapsed Time (Hrs)	Pressure (mm Hg)	Thermocouple Temp. (°F)	Thermocouple ϵ_{th}	Optical Pyrometer Temp. (°F)	Optical Pyrometer ϵ_{th}
1	0.6	1.8×10^{-6}	800	0.807		
	0.8	1.8×10^{-6}	1000	0.805		
	1.0	2.0×10^{-6}	1200	0.866		
	1.2	2.1×10^{-6}	1400	0.904		
	1.5	2.0×10^{-6}	1500	0.915	1499	0.917
	1.7	1.5×10^{-6}	1300	0.909		
	1.9	1.2×10^{-6}	1100	0.894		

TABLE 12

Total Hemispherical Emittance

Coating: Calcium Titanate-Plasma Arc Sprayed (5-Mil)
Substrate: Columbium - 1% Zirconium

Run Number	Elapsed Time (Hrs)	Endurance Time (Hrs)	Pressure (mm Hg)	Thermocouple Temp. (°F)	Thermocouple ϵ_{th}	Avg. ϵ_{th}	Optical Pyrometer Temp (°F)	Optical Pyrometer ϵ_{th}
1	0.5		2.1×10^{-7}	800	0.822			
	1.3		7.6×10^{-7}	901	0.812			
	2.1	0	7.7×10^{-7}	1000	0.812			
	2.4	0.3	4.0×10^{-7}	1000	0.823			
	3.5	1.4	1.6×10^{-7}	1000	0.837			
	4.6	2.5	1.6×10^{-7}	1000	0.847			
	5.5	3.4	1.1×10^{-7}	1000	0.850			
	6.4	4.3	6.0×10^{-8}	1000	0.856			
	7.2	5.1	6.0×10^{-8}	1000	0.858			
	23.8	21.7	3.4×10^{-8}	1000	0.870			
	26.0	23.9	2.0×10^{-8}	1000	0.871	0.871		
	28.1	26.0	2.0×10^{-8}	1000	0.871			
	30.4	28.3	2.0×10^{-8}	1000	0.871			
	48.5	46.4	1.4×10^{-8}	1000	0.872			
	51.4	49.3	1.4×10^{-8}	1000	0.872	0.872		
	53.4	51.3	1.4×10^{-8}	1000	0.872			
	55.3	53.2	1.4×10^{-8}	1000	0.872			
	120.2	118.1	2.8×10^{-8}	1000	0.872			
	122.4	120.3	1.4×10^{-8}	1000	0.872	0.872		
	124.3	122.2	1.3×10^{-8}	1000	0.872			
	126.2	124.1	1.3×10^{-8}	1000	0.872			
	126.4		1.2×10^{-8}	800	0.876			
2	126.7		1.3×10^{-8}	1000	0.872			
	127.0	0	2.0×10^{-8}	1100	0.872			
	127.2	0.2	2.0×10^{-8}	1100	0.881			
	143.6	16.6	1.0×10^{-8}	1100	0.874			
	145.6	18.6	1.1×10^{-8}	1100	0.874			
	151.0	24.0	1.0×10^{-8}	1100	0.882			
	167.6	40.6	1.0×10^{-8}	1100	0.883			
	175.3	48.3	9.0×10^{-9}	1100	0.883	0.878		
	192.9	65.9	8.0×10^{-9}	1100	0.883			
	197.9	70.9	8.0×10^{-9}	1100	0.883			
	215.5	88.5	9.0×10^{-9}	1100	0.872			
	216.0	89.0	8.0×10^{-9}	1100	0.878			
	220.4	93.4	7.0×10^{-9}	1100	0.878			
	287.5	160.5	7.0×10^{-9}	1100	0.875			
3	288.0		6.0×10^{-9}	800	0.882			
	288.5		8.5×10^{-9}	1000	0.878			
	288.7		7.0×10^{-9}	1100	0.876			
	289.2	0	1.0×10^{-8}	1200	0.878			
	291.2	2.0	1.0×10^{-8}	1200	0.877			
	293.6	4.4	8.5×10^{-9}	1200	0.877			
	295.1	5.9	8.5×10^{-9}	1200	0.877	0.877		
	312.2	23.0	7.0×10^{-9}	1200	0.878			
	315.4	26.2	7.0×10^{-9}	1200	0.877			
	317.8	28.6	7.0×10^{-9}	1200	0.878			
	318.1	0	2.0×10^{-8}	1300	0.877			
	319.2	1.1	1.8×10^{-8}	1300	0.878	0.877		
	335.8	17.7	9.5×10^{-9}	1300	0.877			
	339.3	21.2	9.5×10^{-9}	1300	0.877			
	340.6	0	3.5×10^{-8}	1400	0.881			
	342.8	2.2	2.5×10^{-8}	1400	0.877			
	343.2	2.6	2.5×10^{-8}	1400	0.877			
	359.5	18.9	1.2×10^{-8}	1400	0.868			
	361.4	20.8	1.2×10^{-8}	1400	0.870			
	363.3	22.7	1.2×10^{-8}	1400	0.864			
	365.1	24.5	1.2×10^{-8}	1400	0.862			
	365.6		3.8×10^{-8}	1500	0.865		1494	0.876
	366.2		1.1×10^{-8}	1300	0.862			
	366.5		9.3×10^{-9}	1100	0.863			
	366.7		8.5×10^{-9}	900	0.867			

TABLE 13

Total Hemispherical Emittance
Coating: Calcium Titanate - Plasma-Arc Sprayed (5-Mil)
Substrate: Columbium-1% Zirconium

Run Number	Elapsed Time (Hrs.)	Endurance Time (Hrs.)	Pressure (mm Hg)	Thermocouple Temp. (°F)	Thermocouple ϵ th	Avg. ϵ th	Optical Pyrometer Temp(°F)	Optical Pyrometer ϵ th
1	0.2		1.6×10^{-8}	300	0.875			
	0.6		3.0×10^{-7}	500	0.876			
	1.0		7.0×10^{-7}	700	0.843			
	1.3		1.7×10^{-6}	900	0.838			
	1.7	0.0	9.2×10^{-7}	1000	0.834			
	2.9	1.2	2.2×10^{-7}	1000	0.835	0.839		
	4.3	2.6	1.3×10^{-7}	1000	0.839			
	6.0	4.3	6.5×10^{-8}	1000	0.846			
	23.5	21.8	2.0×10^{-8}	1000	0.858			
						0.859		
	30.7	29.0	2.2×10^{-8}	1000	0.860			
	47.6	45.9	1.3×10^{-8}	1000	0.855	0.856		
	52.8	51.1	1.4×10^{-8}	1000	0.857			
	54.6	52.9	1.4×10^{-8}	999	0.859			
	71.3	69.6	1.1×10^{-8}	999	0.853	0.855		
	76.1	74.4	1.1×10^{-8}	999	0.853			
	95.3	93.6	1.1×10^{-8}	999	0.856	0.856		
	102.6	100.9	1.1×10^{-8}	999	0.857			
	168.0	166.3	7.0×10^{-9}	999	0.861	0.859		
	175.0	173.3	7.0×10^{-9}	1001	0.857			
	191.4	189.7	6.0×10^{-9}	1001	0.853			
	191.8		5.0×10^{-9}	799	0.867			
	192.2		4.0×10^{-9}	601	0.870			
2	192.5		5.0×10^{-9}	800	0.864			
	192.8		5.5×10^{-9}	1000	0.855			
	193.1	0.0	1.5×10^{-8}	1100	0.856			
	194.1	1.0	1.8×10^{-8}	1101	0.855			
	195.0	1.9	1.9×10^{-8}	1099	0.858	0.857		
	198.8	5.7	1.9×10^{-8}	1100	0.858			
	215.4	22.3	1.5×10^{-8}	1100	0.858			
	216.7	0.0	6.0×10^{-8}	1199	0.863			
	218.9	2.2	5.1×10^{-8}	1200	0.862			
	222.5	5.8	4.7×10^{-8}	1200	0.862	0.863		
	239.5	22.8	3.5×10^{-8}	1200	0.866			
	240.8	0.0	1.7×10^{-7}	1300	0.866			
	242.0	1.2	2.0×10^{-7}	1300	0.867			
	246.7	5.9	1.3×10^{-7}	1300	0.867			
	263.4	22.6	6.2×10^{-8}	1300	0.865			
	267.0	26.2	6.2×10^{-8}	1300	0.865			
	270.6	29.8	6.0×10^{-8}	1300	0.866	0.866		
	335.8	95.0	2.6×10^{-8}	1300	0.865			
	339.7	98.9	3.1×10^{-8}	1300	0.866			
	342.5	101.7	3.0×10^{-8}	1300	0.865			
	359.1	118.3	2.2×10^{-8}	1300	0.866			
	359.9	0.0	6.8×10^{-8}	1400	0.865			
	363.0	3.1	9.0×10^{-8}	1400	0.865	.865		
	366.9	7.0	9.1×10^{-8}	1400	0.863			
	383.3	23.4	6.2×10^{-8}	1400	0.858			
	383.8	0.0	4.8×10^{-7}	1500	0.858			
	388.5	4.7	1.9×10^{-7}	1500	0.833			
	390.9	7.1	1.6×10^{-7}	1500	0.833			
	407.7	23.9	4.0×10^{-8}	1500	0.730			
	411.5	27.7	3.2×10^{-8}	1500	0.722		1499	0.723
	412.7		1.7×10^{-8}	1400	0.720			
	413.0		1.2×10^{-8}	1300	0.719			
	413.2		9.5×10^{-9}	1200	0.720			
	413.5		7.7×10^{-9}	1100	0.724			
	413.8		6.0×10^{-9}	1000	0.535			

TABLE 14

Total Hemispherical Emittance

Coating: Iron Titanate - Plasma-Arc Sprayed (5-mil)
Substrate: Columbium - 1% Zirconium

Run Number	Elapsed Time (Hrs.)	Pressure (mm Hg)	Thermocouple Temp. (°F)	Thermocouple ϵ th	Optical Pyrometer Temp. (°F)	Optical Pyrometer ϵ th
1	0.2	9.1×10^{-7}	1000	0.872		
	0.5	5.5×10^{-7}	1100	0.872		
	0.7	4.6×10^{-7}	1199	0.874		
	1.0	4.4×10^{-7}	1300	0.872		
	1.3	3.3×10^{-7}	1401	0.871		
	1.7	2.2×10^{-7}	1501	0.871	1516	0.843
	2.9	2.5×10^{-7}	1600	0.866	1606	0.856
	3.3	2.4×10^{-7}	1700	0.869	1704	0.863
	3.5	2.6×10^{-7}	1800	0.872	1805	0.865
	4.1	2.7×10^{-7}	1903	0.869	1910	0.859
	4.5	4.8×10^{-7}	2001	0.870	2029	0.831
	4.7	1.1×10^{-6}	2099	0.886	2113	0.867
	4.9	8.9×10^{-6}	2180	0.911	2174	0.919

TABLE 15

Total Hemispherical Emittance
Coating: Iron Titanate (2 Mil)
Substrate: Columbium -1% Zirconium

Run Number	Elapsed Time (Hrs.)	Pressure (mm Hg)	Thermocouple Temp. (°F)	Thermocouple ϵ_{th}	Optical Pyrometer Temp. (°F)	Optical Pyrometer ϵ_{th}
1	0.1	1.5×10^{-6}	800	0.869		
	0.3	1.5×10^{-6}	1000	0.866		
	1.1	1.6×10^{-6}	1200	0.878		
	1.3	1.6×10^{-6}	1300	0.882		
	1.4	1.5×10^{-6}	1400	0.884		
	1.6	1.5×10^{-6}	1500	0.887	1506	0.876
	1.8	1.5×10^{-6}	1600	0.887	1606	0.876
	2.1	1.4×10^{-6}	1700	0.873	1706	0.863
	2.3	1.4×10^{-6}	1800	0.889	1804	0.882
	2.4	1.4×10^{-6}	1700	0.888	1706	0.878
	2.6	1.4×10^{-6}	1550	0.887	1558	0.873
	2.8	1.4×10^{-6}	1250	0.880		
	2.9	1.4×10^{-6}	950	0.864		

TABLE 16

Total Hemispherical Emittance

Coating: Iron Titanate (4-Mil)
Substrate: Columbium - 1% Zirconium

Run Number	Elapsed Time (Hrs.)	Pressure (mm Hg)	Thermocouple Temp. (°F)	Thermocouple ϵ_{th}	Optical Pyrometer Temp. (°F)	Optical Pyrometer ϵ_{th}
1	0.2	1.7×10^{-6}	700	0.863		
	0.4	2.1×10^{-6}	800	0.865		
	0.6	2.1×10^{-6}	900	0.859		
	0.9	2.9×10^{-6}	1001	0.857		
	1.2	2.2×10^{-6}	1099	0.866		
	1.9	4.7×10^{-7}	1199	0.864		
	2.2	1.0×10^{-6}	1300	0.867		
	2.6	1.5×10^{-6}	1400	0.870		
	3.1	3.6×10^{-6}	1500	0.869	1502	0.866
	3.5	3.0×10^{-6}	1600	0.869	1601	0.868
	3.9	5.6×10^{-6}	1700	0.872	1700	0.872
	4.3	7.8×10^{-6}	1800	0.873	1800	0.873
	5.1	4.0×10^{-6}	1800	0.871	1800	0.871
	5.9	2.4×10^{-6}	1800	0.872	1800	0.872

Test terminated because of insufficient cooling.

TABLE 17

Total Hemispherical Emittance

Coating: Iron Titanate-Plasma Arc Sprayed (4-Mil)
Substrate: Columbium - 1% Zirconium

Run Number	Elapsed Time (Hrs)	Endurance Time (Hrs)	Pressure (mm Hg)	Thermocouple Temp. (°F)	Thermocouple ϵ_{th}	Thermocouple Avg ϵ_{th}	Optical Pyrometer Temp. (°F)	Optical Pyrometer ϵ_{th}	Optical Pyrometer Avg. ϵ_{th}
1	11.2		6.9×10^{-7}	701	0.847				
	11.4		7.6×10^{-7}	801	0.844				
	11.7		1.5×10^{-6}	901	0.842				
	11.9		3.5×10^{-6}	1001	0.842				
	12.2		7.8×10^{-6}	1099	0.840				
	13.0		3.5×10^{-6}	1201	0.840				
	13.3		4.6×10^{-6}	1301	0.837				
	13.6		4.8×10^{-6}	1401	0.840				
	13.9		4.8×10^{-6}	1500	0.836		1500	0.836	
	14.3		7.0×10^{-6}	1600	0.844		1603	0.839	
	14.9		7.2×10^{-6}	1700	0.831		1702	0.828	
	15.4	0	8.7×10^{-6}	1799	0.825		1800	0.823	
	16.2	0.8	4.2×10^{-6}	1799	0.824	0.825	1801	0.821	0.822
	16.9	1.5	3.0×10^{-6}	1798	0.825		1802	0.821	
	33.7	18.3	6.8×10^{-7}				1801	0.819	
	36.0	20.6	5.7×10^{-7}				1800	0.815	0.816
	40.4	25.0	5.4×10^{-7}				1802	0.814	
	57.7	42.3	3.8×10^{-7}				1798	0.816	
	61.6	46.2	3.7×10^{-7}				1798	0.817	0.817
	64.8	49.4	3.6×10^{-7}				1800	0.818	
	81.5	66.1	3.0×10^{-7}				1798	0.817	0.818
	88.2	72.8	3.0×10^{-7}				1798	0.818	
	105.7	90.3	2.7×10^{-7}				1798	0.816	0.817
	112.7	97.3	2.6×10^{-7}				1798	0.818	
	178.9	163.5	1.7×10^{-7}				1800	0.822	0.822
	184.2	168.8	1.7×10^{-7}				1800	0.821	
	202.5	187.1	1.6×10^{-7}				1798	0.826	
	202.9		5.0×10^{-8}				1605	0.828	
	203.1		3.7×10^{-8}						
	203.4		3.0×10^{-8}						
	203.8		2.5×10^{-8}						

Note: Thermocouple values unreliable after 16.9 hours and therefore are not reported.

TABLE 18

Total Hemispherical Emittance
Coating: Iron Titanate (3-Mil)
Substrate: Columbium-1% Zirconium

Run Number	Elapsed Time (Hrs.)	Endurance Time (Hrs.)	Pressure (mm Hg)	Temp. °F	∈th	Avg. ∈th	Optical Pyrometer Temp. °F	Optical Pyrometer ∈th	Optical Pyrometer Avg. ∈th
1	0.3		6.7×10^{-7}	800	0.866				
	0.6		1.2×10^{-6}	1000	0.860				
	0.8		2.8×10^{-6}	1200	0.864				
	1.1		5.4×10^{-6}	1400	0.871				
	1.3		3.1×10^{-6}	1600	0.871		1608	0.858	
	2.0	0.0	4.0×10^{-6}	1800	0.883		1808	0.871	
	2.5	0.5	2.0×10^{-6}	1800	0.884		1808	0.871	
	3.3	1.3	1.3×10^{-6}	1800	0.883		1806	0.874	
	5.0	3.0	9.4×10^{-7}	1800	0.885		1807	0.874	
	6.4	4.4	9.3×10^{-7}	1800	0.884		1807	0.873	
			Specimen removed from chamber to reattach voltage lead						
2	0.4		1.1×10^{-7}	800	0.866				
	0.6		3.0×10^{-7}	1000	0.864				
	1.1		1.8×10^{-6}	1200	0.867				
	1.4		8.2×10^{-6}	1400	0.875				
	1.7		4.6×10^{-6}	1600	0.882		1624	0.842	
	2.0	0.0	5.0×10^{-6}	1800	0.887		1824	0.850	
	2.8	0.8	1.6×10^{-6}	1800	0.888		1823	0.853	
	3.8	1.8	1.1×10^{-6}	1800	0.888		1823	0.853	
	4.8	2.8	7.8×10^{-7}	1800	0.891		1822	0.857	
	6.2	4.2	7.0×10^{-7}	1800	0.891		1822	0.854	
	72.3	70.3	1.6×10^{-7}	1800	0.924		1838	0.864	
	75.9	73.9	1.4×10^{-7}	1768	0.922	0.924	1807	0.860	0.861
	78.2	76.2	1.3×10^{-7}	1768	0.922		1808	0.858	
	79.6	77.6	1.3×10^{-7}	1768	0.926		1809	0.861	
	96.1	94.1	1.1×10^{-7}	1769	0.933		1809	0.868	
	99.8	97.8	1.1×10^{-7}	1768	0.932	0.934	1808	0.868	0.869
	103.1	101.1	1.1×10^{-7}	1768	0.937		1809	0.871	
	120.0	118.0	1.1×10^{-7}	1768	0.943		1809	0.877	
	124.0	122.0	1.1×10^{-7}	1768	0.944	0.943	1809	0.877	0.877
	126.8	124.8	1.1×10^{-7}	1768	0.943		1809	0.877	
	144.0	142.0	1.0×10^{-7}	1759	0.949		1807	0.871	
	147.8	145.8	1.0×10^{-7}	1760	0.947	0.947	1807	0.870	0.870
	150.8	148.8	1.0×10^{-7}	1762	0.946		1809	0.870	
	168.0	166.0	1.2×10^{-7}	1759	0.950		1810	0.867	
	171.9	169.9	1.0×10^{-7}	1759	0.951	0.951	1810	0.868	0.868
	175.4	173.4	1.0×10^{-7}	1758	0.953		1810	0.868	

TABLE 19

Total Hemispherical Emittance

Coating: Nickel Chrome Spinel - Plasma Arc Sprayed (2-mil)
Substrate: Columbium - 1% Zirconium

Run Number	Elapsed Time (Hrs.)	Pressure (mm Hg)	Thermocouple Temp. (°F)	Thermocouple ϵ th	Optical Pyrometer Temp. (°F)	Optical Pyrometer ϵ th
1	0.7	1.5×10^{-7}	300	0.786		
	0.9	1.2×10^{-7}	500	0.839		
	1.2	1.2×10^{-7}	700	0.865		
	1.4	2.5×10^{-7}	900	0.874		
	1.5	2.8×10^{-7}	1000	0.873		
	2.0	3.6×10^{-7}	1100	0.878		
	2.1	4.9×10^{-7}	1200	0.876		
	2.3	4.8×10^{-7}	1300	0.878		
	2.6	4.2×10^{-7}	1400	0.879		
	3.6	4.2×10^{-7}	1500	0.871	1500	0.871
	3.8	5.4×10^{-7}	1600	0.870	1601	0.869
	4.1	1.0×10^{-7}	1700	0.867	1701	0.865
	4.3	1.4×10^{-6}	1800	0.866	1807	0.855
	4.5	1.4×10^{-6}	1900	0.868	1906	0.859
	4.7	1.6×10^{-6}	2000	0.867	2003	0.863
	4.9	1.6×10^{-6}	2100	0.878	2097	0.881
	5.1	2.2×10^{-6}	2200	0.878	2189	0.893
	5.2	8.4×10^{-6}	2150	0.883	2148	0.885
	5.4	1.0×10^{-6}	1850	0.880	1861	0.864
	5.6	6.2×10^{-7}	1550	0.872		

TABLE 20

Total Hemispherical Emittance

Coating: Nickel-Chrome Spinel - Plasma-Arc Sprayed (4-Mil)
Substrate: Columbium-1% Zirconium

Run Number	Elapsed Time(Hrs.)	Pressure (mm Hg)	Thermocouple Temp.(°F)	Thermocouple ϵ th	Optical Pyrometer Temp.(°F)	Optical Pyrometer ϵ th
1	0.2	1.4×10^{-6}	800	0.909		
	0.4	1.5×10^{-6}	1000	0.908		
	0.7	2.0×10^{-6}	1200	0.910		
Rig Opened; Specimen Removed and Later Reinstalled						
2	0.1	1.2×10^{-6}	800	0.891		
	0.3	1.1×10^{-6}	1000	0.925		
	0.4	1.3×10^{-6}	1200	0.899		
	1.0	2.5×10^{-6}	1400	0.889		
	1.2	8.0×10^{-6}	1600	0.898	1608	0.884
	1.4	2.1×10^{-5}	1800	0.906	1817	0.879
	1.6	1.3×10^{-5}	1700	0.923		

TABLE 21

Total Hemispherical Emittance

Coating: Nickel-Chrome Spinel-Plasma-Arc Sprayed (4-Mil)
Substrate: Columbium - 1% Zirconium

Run Number	Elapsed Time (Hrs)	Endurance Time (Hrs)	Pressure (mm Hg)	Thermocouple Temp. (°F)	Thermocouple ϵ_{th}	Optical Pyrometer Temp. (°F)	Optical Pyrometer ϵ_{th}
1	0.5		3.0×10^{-7}	999	0.678		
	0.6		1.0×10^{-8}	1100	0.689		
	0.65		5.0×10^{-7}	1199	0.703		
	0.7		3.0×10^{-7}	1300	0.711		
	0.8		3.0×10^{-7}	1400	0.717		
	0.9		5.0×10^{-7}	1500	0.724	1497	0.728
	1.1		7.0×10^{-7}	1600	0.722	1599	0.724
	1.2		1.5×10^{-6}	1701	0.724	1704	0.720
	12.6	0.0	2.0×10^{-6}	1800	0.722	1800	0.722
	13.5	0.9	9.2×10^{-7}	1800	0.704	1799	0.705
	14.0	1.4	6.6×10^{-7}	1800	0.704	1800	0.704
	16.0	3.4	2.7×10^{-7}	1800	0.681	1802	0.679
	18.0	5.4	2.2×10^{-7}	1800	0.670	1801	0.669
	19.0	6.4	1.4×10^{-7}	1800	0.665	1801	0.664
	28.3	15.7	6.0×10^{-7}	1800	0.690	1813	0.675
	30.1	17.5	5.0×10^{-7}	1800	0.675	1805	0.669
	30.9		2.0×10^{-8}	1650	0.660	1650	0.660
	32.8		1.4×10^{-8}	1451	0.639	1467	0.618
	33.5		8.0×10^{-9}	1251	0.603		
	33.9		8.0×10^{-9}	1049	0.570		

TABLE 22

Total Hemispherical Emittance
Coating: Nickel Chrome Spinel (4-Mil)
Substrate: Columbium-1% Zirconium

Run Number	Elapsed Time (Hrs.)	Endurance Time (Hrs.)	Pressure (mm Hg)	Thermocouple Temp. °F	Thermocouple ϵ th	Thermocouple Avg. ϵ th	Optical Pyrometers Temp. °F	Optical Pyrometers ϵ th	Optical Pyrometers Avg. ϵ th
1	0.5		5.4×10^{-7}	800	0.896				
	0.6		1.3×10^{-6}	1000	0.882				
	0.9		1.7×10^{-6}	1200	0.885				
	1.1		2.6×10^{-6}	1400	0.887				
	2.4	1.0	8.5×10^{-7}	1600	0.885		1599	0.886	
	3.3	1.9	5.7×10^{-7}	1600	0.885	0.885	1599	0.886	0.886
	7.2	5,8	3.0×10^{-7}	1600	0.884		1599	0.886	
	23.4	22.0	1.3×10^{-7}	1600	0.885		1603	0.880	
	28.4	27.0	1.1×10^{-7}	1600	0.885	0.885	1603	0.880	0.880
	30.4	29.0	1.1×10^{-7}	1600	0.885		1603	0.880	
	96.0	94.6	4.0×10^{-8}	1600	0.883		1604	0.876	
	99.4	98.0	4.4×10^{-8}	1600	0.883	0.883	1604	0.876	0.876
	103.1	101.7	4.7×10^{-8}	1600	0.883		1604	0.877	
	120.1	118.7	3.5×10^{-8}	1600	0.883		1604	0.876	
	123.3	121.9	3.9×10^{-8}	1600	0.883	0.883	1604	0.876	0.876
	126.4	125.0	4.0×10^{-8}	1600	0.883		1604	0.876	
	143.5	142.1	3.5×10^{-8}	1600	0.883		1604	0.876	
	147.5	146.1	3.6×10^{-8}	1600	0.883	0.883	1604	0.877	0.876
	151.2	149.8	3.7×10^{-8}	1600	0.883		1604	0.876	
	167.9	166.5	3.1×10^{-8}	1600	0.888		1604	0.881	
	171.5	170.1	3.4×10^{-8}	1600	0.887	0.887	1604	0.880	0.880
	175.2	173.8	3.5×10^{-8}	1600	0.887		1604	0.880	
	192.8	191.4	3.0×10^{-8}	1593	0.895		1604	0.876	
	195.9	194.5	3.1×10^{-8}	1595	0.893	0.894	1604	0.878	0.877
	198.8	197.4	3.4×10^{-8}	1594	0.893		1604	0.876	
	263.8	262.4	2.1×10^{-8}	1589	0.901		1604	0.875	
	265.9		1.4×10^{-8}	1395	0.888				
	266.2		1.1×10^{-8}	1197	0.879				
	266.5		9.8×10^{-9}	997	0.874				

TABLE 23

Total Hemispherical Emittance
Coating: Nickel Chrome Spinel
Substrate: Columbium-1% Zirconium

Run Number	Elapsed Time (Hrs.)	Endurance Time (hrs.)	Pressure (mm Hg)	Thermocouple Temp. (°F)	Thermocouple ϵ th	Optical Pyrometer Temp. (°F)	Optical Pyrometer ϵ th
1	0.4		2.1×10^{-7}	.800	0.863		
	0.6		2.2×10^{-7}	900	0,868		
	0.8		2.1×10^{-7}	1000	0.861		
	1.2		3.1×10^{-7}	1100	0.870		
	1.4		4.3×10^{-7}	1200	0.868		
	1.7		8.5×10^{-7}	1301	0.874		
	2.3		1.7×10^{-6}	1400	0.872		
	2.8		3.0×10^{-6}	1500	0.875	1496	0.883
	3.1		8.0×10^{-6}	1600	0.874	1599	0.876
	3.5		6.2×10^{-6}	1700	0.877	1704	0.870
	3.9	0	8.0×10^{-6}	1800	0.878	1806	0.869
	5.1	1.2	2.6×10^{-6}	1800	0.832	1808	0.820
	71.0	67.1	2.4×10^{-7}	1800	0.875	1830	0.830
	74.5	70.6	3.5×10^{-7}			1830	0.829
	77.4	73.5	3.5×10^{-7}			1830	0.841
	98.5	94.6	2.2×10^{-7}			1832	0.839
	98.7	94.8	5.0×10^{-8}			1547	0.835

TABLE 24

Total Hemispherical Emittance
Coating: Silicon Carbide (7-Mil)
Substrate: Columbium -1% Zirconium

Run Number	Elapsed Time (Hrs.)	Pressure (mm Hg)	Thermocouple Temp. (°F)	ϵ th
1	1.0	2.0×10^{-6}	300	0.862
	1.3	2.0×10^{-6}	500	0.836
	1.6	2.0×10^{-6}	700	0.846
	1.9	2.3×10^{-6}	900	0.823
	2.2	3.4×10^{-6}	1000	0.828
	2.4	3.8×10^{-6}	1100	0.839
	3.4	2.3×10^{-6}	1200	0.809
	3.6	2.3×10^{-6}	1300	0.796

TABLE 25

Total Hemispherical Emittance
Coating: Silicon Carbide (4-Mil)
Substrate: Columbium -1% Zirconium

Run Number	Elapsed Time (Hrs.)	Pressure (mm Hg)	Thermocouple Temp. (°F)	Thermocouple ϵ th	Optical Pyrometer Temp. (°F)	Optical Pyrometer ϵ th
1	0.9	1.6×10^{-6}	300	0.829		
	1.2	1.6×10^{-6}	500	0.863		
	1.4	1.6×10^{-6}	700	0.872		
	1.6	2.0×10^{-6}	900	0.863		
	1.8	2.7×10^{-6}	1000	0.844		
	2.0	3.4×10^{-6}	1100	0.863		
	2.2	3.0×10^{-6}	1200	0.840		
	2.3	2.6×10^{-6}	1300	0.845		
	2.5	2.4×10^{-6}	1402	0.839		
	3.5	2.0×10^{-6}			1485	0.867

TABLE 26

Total Hemispherical Emittance
Coating: Enamel (2-Mil)
Substrate: AISI-310 Stainless Steel

Run Number	Elapsed Time (Hrs.)	Pressure (mm Hg)	Thermocouple Temp. (°F)	Thermocouple ϵ_{th}	Optical Pyrometer Temp. (°F)	Optical Pyrometer ϵ_{th}
1	0.1	1.5×10^{-6}	800	0.838		
	0.3	1.5×10^{-6}	900	0.841		
	0.4	1.4×10^{-6}	1000	0.847		
	0.8	1.4×10^{-6}	1100	0.858		
	1.1	1.5×10^{-6}	1200	0.858		
	1.3	1.5×10^{-6}	1400	0.871		
	1.5	1.5×10^{-6}	1500	0.874		
	1.6	1.5×10^{-6}	1550	0.867	1554	0.860
	1.9	1.5×10^{-6}	1600	0.886	1600	0.886
2	2.0	1.5×10^{-6}	1400	0.876		
	2.1	1.5×10^{-6}	1500	0.865	1504	0.858
	2.2	1.5×10^{-6}	1600	0.891	1598	0.894
	2.3	1.4×10^{-6}	1650	0.895	1651	0.893
	2.4	1.5×10^{-6}	1700	0.892	1699	0.893

TABLE 27

Residual Gas Analysis Results for Long Term Endurance Rig Number 1

Analysis	M/ne	Concentration Ratio	Absolute Pressure (mm Hg) Background	Rig
H_2	2	0.242	34.18×10^{-11}	47.28×10^{-11}
CO, CO_2	12	0.227		
Ar	13	0.308		
N_2	14	0.298		
CH_4	15	0.287		
O_2, CH_4, CO, CO_2	16	0.350		
NH_3, H_2O	17	0.526		
H_2O	18	0.532		
F, OH_3^+	19	0.168		
Ne, Ar	20	1.181		
C_2H_5	25	0.394		
C_2H_5	26	0.302		
C_2H_3	27	0.240		
N_2CO	28	0.265	31.95×10^{-11}	48.40×10^{-11}
C_2H_5, C_3H_8, N_2	29	0.274		
NO, C_2H_6	30	0.350		
O_2, S, Methanol	32	0.438	1.80×10^{-11}	4.51×10^{-11}
H_2S, O_2	34	0.394		
Cl	35	0.214		
HCl, Cl	36	0.262		
Cl	37	0.320		
HCl	38	0.328		
Ar	40	1.292	1.12×10^{-11}	8.91×10^{-11}
Olefins, C_3H_8	41	0.243		
Propylene, C_3H_8	42	0.302		
Paraffins, C_3H_8	43	0.320		
CO_2, C_3H_8	44	0.220	3.50×10^{-11}	4.43×10^{-11}

Notes: Rig number 1 contained a SNAP-8 test section coated with a mixture of nickel-chrome spinel and silicon dioxide

Concentration ratio equals (rig deflection/background deflection) X (background pressure/rig pressure) and represents ratio of final concentration to initial concentration

M/ne equals nuclear mass per unit charge

Exposure time: 15,000 hours

TABLE 28

Results of Spectrographic and X-Ray Analyses of Long Term Endurance Test Sections

Spectrographic Analysis

Specimen		
	1	Al, Cr, Si, Ni, P
	2	Ti, Al
	3	Ti, Al
	4	Al, Cr, Si, P

X-Ray Fluorescence

Specimen		
	1	Cr, Ni, Si, Fe, Zn
	2	Ti, Fe, Zn, Cr
	3	Ti, Fe, Ni, Zn
	4	Cr, Si, Fe, Zn, Ni

X-Ray Diffraction

Specimen		
	1	$AlPO_4$, SiO_2, $NiCr_2O_4$, SiC
	2	TiO_2
	3	TiO_2
	4	$AlPO_4$, SiO_2, SiC

Note: All constituents listed in order of decreasing estimated concentrations

TABLE 29

Results of Hardness Testing of Long Term Endurance Test Sections

Specimens	Location*	Hardness (Vickers Hardness)
1	A	37.4, 36.8
	B	31.9, 29.0
	C	25.6, 25.6, 28.4
	D	24.1
	E	26.8, 24.1, 26.0
	F	24.5
	G	24.9, 24.1, 23.5
2	A	33.5, 37.4
	B	39.3, 31.9
	C	29.6, 28.0, 28.8
	D	27.2
	E	26.6, 27.8, 25.4
	F	22.9
	G	24.4, 25.8, 26.1
3	H	174, 165
	I	46.8, 42,8, 39.0
	J	32.9
	K	33.5
	L	31.6
4	A	37.7, 36.2
	B	35, 27.8
	C	25.5, 24.7, 25.8
	D	25.8
	E	25.8, 25.4, 25.2
	F	29.2
	G	25.2, 26.6, 26.0

* See Figures 39 and 55 for test locations.

TABLE 30

Results of Mechanical Testing of
Long Term Endurance Test Sections

Endurance Test Number	Location	Bond Strength (psi)	Bend Angle to Failure* (Degrees)	Form of Coating Failure During Bend Testing
1	Root	5480	98	edge spalling
1	Tip	3300	142	very small cracks
2	Root	7050	65	cracks
2	Tip	9820	48	spalling
3	Root	8500	43	cracks and spalling
3	Tip	5700	62	cracks and spalling
4	Root	4820	72	powdered
4	Tip	6500	90	powdered

* Specimens bent over mandrel five times the material thickness.

TABLE 31

Residual Gas Analysis Results for Long Term Endurance Rig Number 2

Analysis	M/ne	Concentration Ratio	Absolute Pressure (mm Hg) Background	Rig
H_2	2	0.311	40.98×10^{-11}	68.26×10^{-11}
CO, CO_2	12	0.256		
Ar	13	0.322		
N_2	14	0.411		
CH_4	15	0.330		
O_2, CH_4, CO, CO_2	16	0.448		
NH_3, H_2O	17	0.666		
H_2O	18	1.647		
F, OH_3^+	19	0.486		
Ne, Ar	20	1.810		
Ne, CO_2	22	0.204		
Complex Hydrocarbons	24	0.374		
C_2H_2	25	0.444		
C_2H_2	26	0.362		
C_2H_3	27	0.279		
N_2, CO	28	0.284	31.84×10^{-11}	48.40×10^{-11}
C_2H_5, C_3H_8, N_2	29	0.306		
NO, C_2H_6	30	0.320		
O_2, S, Methanol	32	0.374	2.05×10^{-11}	4.10×10^{-11}
H_2S	33	0.262		
H_2S, O_2	34	0.327		
Cl	35	0.186		
HCl, Cl	36	0.220		
Cl	37	0.249		
HCl	38	0.320		
Dienes, Ar	39	0.287		
Ar	40	2.020	0.85×10^{-11}	9.18×10^{-11}
Olefins, C_3H_8	41	0.262		
Propylene, C_3H_8	42	0.254		
Paraffins, C_3H_8	43	0.267		
CO_2, C_3H_8	44	0.213	4.60×10^{-11}	5.25×10^{-11}

Notes: Rig number 2 contained a SNAP-8 test section coated with Titania Base

Concentration ratio equals (rig deflection/background deflection) X (background pressure/rig pressure) and represents ratio of final concentration to initial concentration

M/ne equals nuclear mass per unit charge

Exposure Time: 14,037 hours

TABLE 32

Residual Gas Analysis Results for Long
Term Endurance Rig Number 3

Analysis	M/ne	Concentration Ratio	Absolute Pressure (mm Hg) Background	Rig
H_2	2	0.510	18.75×10^{-11}	3.18×10^{-11}
CO, CO_2	12	0.800		
Ar	13	0.812		
N_2	14	0.733		
CH_4	15	0.806		
O_2, CH_4, CO, CO_2	16	1.073		
NH_3, H_2O	17	0.786		
H_2O	18	0.762		
F, OH_3^+	19	1.020		
C_2H_5	25	1.714		
C_2H_5	26	0.761		
C_2H_3	27	1.923		
N_2, CO	28	2.532	17.44×10^{-11}	14.72×10^{-11}
C_2H_5, C_3H_8, N_2	29	1.000		
O_2, S, Methanol	32	2.600	0.25×10^{-11}	2.13×10^{-11}
HCl	38	6.000		
Dienes, Ar	39	0.770		
Ar	40	2.083	0.99×10^{-11}	6.85×10^{-11}
Olefins, C_3H_8	41	0.688		
Propylene, C_3H_8	42	1.800		
Paraffins, C_3H_8	43	7.714		
CO_2, C_3H_8	44	0.800	0.53×10^{-11}	1.42×10^{-11}

Notes: Rig number 3 contained a Sunflower I test section coated with Titania Base

Concentration Ratio equals (rig deflection/background deflection)X (background pressure/rig pressure) and represents ratio of final concentration to initial concentration

M/ne equals nuclear mass per unit charge

Exposure time: 13,755 hours

TABLE 33
Residual Gas Analysis Results for Long Term Endurance Rig Number 4

Analysis	M/ne	Concentration Ratio	Absolute Pressure (mm Hg) Background	Rig
H_2	2		3.70×10^{-10}	
CO, CO_2	12	0.323		
Ar	13	0.399		
N_2	14	0.473		
CH_4	15	0.436		
O_2, CH_4, CO, CO_2	16	0.450		
NH_3, H_2O	17	0.890		
H_2O	18	0.912		
F, OH_3^+	19	0.833		
Ne, Ar	20	2.214		
Ne	21			
Ne, CO_2	22	0.296		
	23			
	24			
C_2H_2	25	0.833		
C_2H_2	26	0.476		
C_2H_3	27	0.430		
N_2, CO	28	0.403	5.75×10^{-10}	6.95×10^{-10}
C_2H_5, C_3H_8, N_2	29	0.413		
NO, C_2H_6	30	0.633		
CH_2OH^+	31			
O_2, S, Methanol	32	0.430	3.12×10^{-11}	4.02×10^{-11}
H_2S	33	0.866		
H_2S, O_2	34	0.483		
Cl	35			
HCl, Cl	36	0.666		
Cl	37	1.066		
HCl	38			
Dienes, Ar	39	0.366		
Ar	40	2.081	1.206×10^{-11}	7.54×10^{-11}
Olefins, C_3H_8	41	0.373		
Propylene, C_3H_8	42	0.376		
Paraffins, C_3H_8	43	0.433		
CO_2, C_3H_8	44	0.316	1.115×10^{-10}	1.056×10^{-10}

Notes: Rig number 4 contained SNAP-8 test section coated with SiC and SiO_2

Concentration ratio equals (rig deflection/background deflection) X (background pressure/rig pressure) and represents ratio of final concentration to initial concentration

M/ne equals nuclear mass per unit charge

Exposure time: 12,781 hours

TABLE 34

Results of Investigation of Alkaphos C-Bonded Coating Procedures

Specimum Number	Coating	Surface Preparation	Substrate	Slurry Composition	Air Drying Time (Hours)	Curing Cycle *	Results	Remarks
1 2 3 4	Black SiC Black SiC Black SiC Black SiC	Chemically Cleaned Vapor Blasted Grit Blasted-90 Grit Al_2O_3 Grit Blasted-28 Grit Steel	Cb-1Zr Cb-1Zr Cb-1Zr Cb-1Zr	100 gms SiC 100 ml Alkaphos	66	180F(1) + 220F(1) + 300F (2)	No change noted after 180F+ 220F cycle. Steel grit blasted tube coating was very rough. After 300F cycle all tubes showed evidence of bubbling with Al_2O_3 grit blasted tube showing the least.	Specimens were furnace cooled from 700F in about four hours. Coatings exhibited crust-like surface (bubbling). After standing in cabinet about 2 hours coatings spalled off of all 4 tubes
						375F(1 1/2) + 700F(3)	More evidence of bubbling after two latter cycles.	
5	Black SiC	Vapor Blasted	Stainless Steel	100 gms SiC 100 ml Alkaphos	2	400F(2) + 900F(2)	No bonding. Light, medium and heavy coatings were powdery after curing.	Medium and heavy coatings showed evidence of bubbling.
6	Black SiC	Vapor Blasted	Stainless Steel	100 gms SiC 100 ml Alkaphos	20	400F(2) + 900F(2)	No bonding. Light, medium and heavy coatings were powdery after curing.	Medium and heavy coatings showed evidence of bubbling.
7	Black SiC	Vapor Blasted	Stainless Steel	100 gms SiC 100 ml Alkaphos	48	300-350F(64)	No bond between coating and substrate after curing. Coating was uniform and could not be rubbed off of substrate after air dry.	Coating came off in one complete sheet. Good bond between particles. Evidence of bubbling.
8	Black SiC	Vapor Blasted	Stainless Steel	100 gms SiC 100 ml Alkaphos	100	180-250F(10) + 500F(2) + 950F(2)	No bonding between coating and substrate.	Coating came off in complete sheet. Good bond between particles. Evidence of bubbling.
9	Black SiC	Vapor Blasted	Stainless Steel	100 gms SiC 100 ml Alkaphos	336	200F(2) + 260F(15) + 300F(2) + 400F(2) + 700F(3)	Coating showed no bubbling during first 4 cycles.	Coating crumbled after cooling and setting in air for 24 hours.
10	Black SiC	Grit Blasted-90 Grit Al_2O_3	Stainless Steel	100 gms SiC 100 ml Alkaphos	24	250F(10)+500F(2)+ 950F(2)	No bond between coating and substrate.	

* Numbers in parenthese indicate hours at temperature

TABLE 34(Cont'd.)

Specimen Number	Coating	Surface Preparation	Substrate	Slurry Composition	Air Drying Time (Hours)	Curing Cycle*	Results	Remarks
11	Black SiC	Grit Blasted-90 Grit Al_2O_3	Cb-1Zr	100 gms SiC 100 ml Alkaphos	24	250F(10) + 500F(2) + 950F(2)	Coating adhered quite well	Substrate oxidized at 900F. Evidence of bubbling.
12	Black SiC	Grit Blasted-90 Grit Al_2O_3	Cb-1Zr	100 gms SiC 100 ml Alkaphos	68	200F(2) + 250F(15)+ 300F(2) + 400F(2) + cooled + 700F(2)	Coating bubbled slightly after 300F cycle. Coating was hard and intact after 700F before furnace cool.	Coating was hard and intact. Could not be scraped from tube except in bubbled areas after 400F cycle.
13	Green SiC	Grit Blasted - 90 Grit Al_2O_3	Cb-1Zr	100 gms SiC 100 ml Alkaphos	20	200F(2) + 250F(2) + 300F(2) + 400F(2) + cooled + 700F(2)	Bubbled slightly after 400F cure. Excessive spalling and breakdown of bond after 700F.	
14	Green SiC	Grit Blasted-90 Grit Al_2O_3	Cb-1Zr	100 gms SiC 100 ml Alkaphos	20	200F(2) + 250F(2) + 300F(2) + 400F(2)	Coating showed slight bubbling after 400F cycle but otherwise exhibited good bonding.	
15	Green SiC	Grit Blasted - 90 Grit Al_2O_3	Cb-1Zr	100 gms SiC 100 ml Alkaphos 10 ml H_3PO_4	20	200F(2) + 250F(2) + 300F(2) + 400F(2) + cooled + 700F(2)	Did not cure during cycles to 400F. Excessive spalling after 700F.	
16.	$SrTiO_2$	Grit Blasted - 90 Grit Al_2O_3	Cb-1Zr	100 gms $SrTiO_3$ 125 ml Alkaphos	20	200F(2) + 250F(2) + 300F(2) + 400F(2) + cooled + 700F(2)	Very strong bond of coating to substrate. No evidence of bubbling.	Coating was white after 400F cycle but had a brownish tinge after 700F cycle. Coating seemed to be more brittle after 700F cure.
17	Alkaphos C	Grit Blasted-90 Grit Al_2O_3	Stainless Steel	No filler	90	250F(10) + 500F(2) + 950F(2)	Coating was white and flaky and could be scraped off. Little evidence of bubbling.	Coating was tacky after drying. Showed areas of incomplete wetting.
18	Alkaphos C	Grit Blasted-90 Grit Al_2O_3	Cb-1Zr	No filler	90	250F(10) + 500F(2) + 950F(2)	Color varied from transparent in thin areas to white in thick areas. Coating could be scraped off but had little bubbling.	Coating was tacky after drying. Showed areas of incomplete wetting.
19	$CaTiO_3$	Grit Blasted-90 Grit Al_2O_3	Cb-1Zr	100 gms $CaTiO_3$ 150 ml Alkaphos	20	200F(2) + 250F(2) + 300F(2) + 400F(2)	Good bond between coating and substrate. Coating turned reddish color on exposed top side. No bubbling.	Coating difficult to apply because of settling of particles in solution.

* Numbers in parentheses indicate hours at temperature

TABLE 35

Temperature Values Obtained With 3-Mil Diameter Thermocouples on Uncoated Polished Tantalum

Platinum-Platinum 10% Rhodium		Chromel-Alumel
Improperly Annealed Condition Temperature Reading (°F)	Fully Annealed Condition Temperature Reading (°F)	As-Received Condition Temperature (°F)
196	200	199
393	400	400
592	601	600
790	800	800
990	1000	1000
1191	1199	1200
1393	1400	1400
1596	1600	1601

TABLE 36

Temperature Values Obtained with Fully Annealed 1- and 3-Mil Diameter Platinum-Platinum 10 Per Cent Rhodium Thermocouples on Uncoated, Polished Tantalum

Temperature Reading with 1-Mil Diameter Wire (°F)	Temperature Reading with 3-Mil Diameter Wire (°F)
999	999
1195	1194
1399	1396
1604	1602
1809	1808
2006	2006
2207	2204

TABLE 37

Total Hemispherical Emittance
Coating: Partially Oxidized Hastelloy C (7-Mil)
Substrate: A I S I -310 Stainless Steel

Run Number	Elapsed Time (Hrs.)	Pressure (mm Hg)	Thermocouple Temp. (°F)	Thermocouple ϵ_{th}	Optical Pyrometer Temp. (°F)	Optical Pyrometer ϵ_{th}
1	0.6	4.4×10^{-6}	327	0.476		
	1.0	4.8×10^{-6}	526	0.493		
	1.4	5.5×10^{-6}	726	0.502		
	3.1	1.4×10^{-6}	922	0.525		
	3.4	1.3×10^{-6}	1024	0.530		
	3.8	1.4×10^{-6}	1124	0.535		
	4.0	6.0×10^{-7}	1226	0.541		
	4.3	5.4×10^{-7}	1328	0.549		
	4.5	5.4×10^{-7}	1428	0.548		
	4.9	6.4×10^{-7}	1530	0.563	1505	0.592
	5.2	7.6×10^{-7}	1632	0.577	1599	0.557
	5.4	2.4×10^{-7}	1735	0.575	1710	0.602
	5.7	5.4×10^{-7}	1276	0.523		
	5.9	4.9×10^{-7}	971	0.524		

TABLE 38

Total Hemispherical Emittance
Coating: Partially Oxidized Hastelloy X (8-Mil)
Substrate: A I S I -310 Stainless Steel

Run Number	Elapsed Time (Hrs.)	Pressure (mm Hg)	Thermocouple Temp. (°F)	Thermocouple ϵ th	Optical Pyrometer Temp. (°F)	Optical Pyrometer ϵ th
1	0.7	1.0×10^{-6}	322	0.522		
	1.2	1.2×10^{-6}	524	0.538		
	1.5	1.2×10^{-6}	726	0.551		
	1.7	1.1×10^{-6}	927	0.575		
	1.9	1.0×10^{-6}	1028	0.581		
	2.1	1.0×10^{-6}	1128	0.589		
	2.4	9.0×10^{-7}	1228	0.585		
	2.7	7.7×10^{-7}	1329	0.602		
	2.9	9.0×10^{-7}	1430	0.611		
	3.1	1.2×10^{-6}	1531	0.619	1513	0.642
	3.3	2.8×10^{-6}	1633	0.627	1614	0.650
	3.5	9.7×10^{-6}	1736	0.636	1727	0.647
	3.6	8.6×10^{-7}	1277	0.606		
	3.8	7.4×10^{-7}	1073	0.587		

TABLE 39

Total Hemispherical Emittance
Coating: Oxidized Kennametal K-151A (4-Mil)
Substrate: A I S I -310 Stainless Steel

Run Number	Elapsed Time (Hrs.)	Pressure (mm Hg)	Thermocouple Temp. (°F)	Thermocouple ϵ th	Optical Pyrometer Temp (°F)	Optical Pyrometer ϵ th
1	1.0	2.1×10^{-6}	529	0.751		
	1.2	1.9×10^{-6}	729	0.784		
	1.4	1.8×10^{-6}	929	0.812		
	1.7	1.7×10^{-6}	1032	0.813		
	2.0	1.7×10^{-6}	1132	0.805		
	2.2	1.7×10^{-6}	1232	0.828		
	2.4	2.0×10^{-6}	1335	0.832		
	2.6	2.7×10^{-6}	1435	0.836		
	3.0	4.2×10^{-6}	1537	0.843	1515	0.881
	3.3	9.9×10^{-6}	1640	0.860	1614	0.905
	3.5	3.4×10^{-5}	1744	0.863	1727	0.890
	3.7	1.2×10^{-6}	973	0.804		
	3.9	1.2×10^{-6}	668	0.745		

TABLE 40

Total Hemispherical Emittance
Coating: Oxidized Kennametal K-162B (5-Mil)
Substrate: A I S I -310 Stainless Steel

Run Number	Elapsed Time (Hrs.)	Pressure (mm Hg)	Thermocouple Temp. (°F)	Thermocouple ϵ th	Optical Pyrometer Temp. (°F)	Optical Pyrometer ϵ th
1	0.3	3.8×10^{-6}	323	0.768		
	0.7	5.7×10^{-6}	525	0.792		
	1.9	8.4×10^{-6}	723	0.820		
	2.2	4.4×10^{-6}	927	0.832		
	2.5	3.4×10^{-6}	1028	0.841		
	2.7	3.9×10^{-6}	1129	0.853		
	2.9	4.0×10^{-6}	1230	0.860		
	3.2	5.0×10^{-6}	1331	0.872		
	3.4	6.9×10^{-6}	1431	0.878		
	3.9	9.6×10^{-6}	1534	0.885	1503	0.942
	4.1	2.3×10^{-5}	1636	0.892	1611	0.936
	4.5	4.3×10^{-5}	1740	0.892	1687	0.984
	4.7	4.6×10^{-6}	1482	0.869		
	4.9	3.7×10^{-6}	1279	0.861		
	5.1	3.7×10^{-6}	1033	0.952		
	5.2	3.7×10^{-6}	872	0.834		

TABLE 41

Total Hemispherical Emittance
Coating: Barium Titanate (7-Mil)
Substrate: Columbium -1% Zirconium

Run Number	Elapsed Time (Hrs.)	Pressure (mm Hg)	Thermocouple Temp. (°F)	ϵ_{th}
1	0.5	1.9×10^{-7}	323	0.853
	0.9	1.8×10^{-7}	545	0.727
	1.5	1.8×10^{-7}	723	0.741
	1.9	2.0×10^{-7}	926	0.662
	2.4	2.3×10^{-7}	1027	0.588
	2.9	2.3×10^{-6}	823	0.639
	3.6	2.2×10^{-6}	618	0.700
	4.0	2.1×10^{-6}	417	0.771

TABLE 42

Total Hemispherical Emittance
Coating: Calcium Titanate (5-Mil)
Substrate: Columbium -1% Zirconium

Run Number	Elapsed Time (Hrs.)	Pressure (mm Hg)	Thermocouple Temp. (°F)	Thermocouple ϵ_{th}	Optical Pyrometer Temp. (°F)	Optical Pyrometer ϵ_{th}
1	0.4	3.1×10^{-6}	330	0.750		
	0.9	6.2×10^{-6}	526	0.738		
	1.4	6.3×10^{-6}	713	0.745		
	1.9	9.5×10^{-6}	923	0.710		
	2.1	8.7×10^{-6}	1024	0.724		
	2.4	8.1×10^{-6}	1122	0.774		
	2.6	8.8×10^{-6}	1222	0.823		
	2.9	6.6×10^{-6}	1319	0.871		
	3.8	5.3×10^{-6}	1423	0.901		
	4.0	5.2×10^{-6}	1519	0.904	1498	0.944
	4.5	5.6×10^{-6}	1272	0.889		
	4.8	5.6×10^{-6}	1022	0.857		
	5.4	7.0×10^{-6}	819	0.859		
2	5.5	4.9×10^{-6}	1518	0.915	1498	0.953
	5.8	4.2×10^{-6}	1617	0.910	1594	0.951
	6.0	4.2×10^{-6}	1418	0.907		
	6.3	4.8×10^{-6}	1221	0.893		

TABLE 43

Total Hemispherical Emittance
Coating: Calcium Titanate (4-Mil)
Substrate: Columbium -1% Zirconium

Run Number	Elapsed Time (Hrs.)	Pressure (mm Hg)	Thermocouple Temp. (°F)	Thermocouple ϵ th	Optical Pyrometer Temp. (°F)	Optical Pyrometer ϵ th
1	1.5	2.1×10^{-6}	321	0.752		
	2.0	1.7×10^{-6}	524	0.765		
	2.5	1.7×10^{-6}	726	0.745		
	2.9	1.6×10^{-6}	927	0.744		
	3.1	2.0×10^{-6}	1026	0.746		
	3.4	2.2×10^{-6}	1126	0.791		
	3.6	2.0×10^{-6}	1225	0.846		
	4.0	1.6×10^{-6}	1325	0.891		
	4.3	1.4×10^{-6}	1429	0.901		
	4.4	1.6×10^{-6}	1533	0.902	1506	0.953
	4.8	1.4×10^{-6}	1286	0.896		

TABLE 44

Total Hemispherical Emittance
Coating: Iron Titanate (4-Mil)
Substrate: Columbium-1% Zirconium

Run Number	Elapsed Time (Hrs.)	Pressure (mm Hg)	Thermocouple Temp. (°F)	Thermocouple ϵ th	Optical Pyrometer Temp. (°F)	Optical Pyrometer ϵ th
1	0.8	7.8×10^{-6}	335	0.790		
	1.4	1.1×10^{-6}	523	0.873		
	1.7	1.8×10^{-6}	722	0.822		
	2.2	2.8×10^{-6}	927	0.818		
	2.7	3.0×10^{-6}	1028	0.794		
	3.0	2.9×10^{-6}	1123	0.785		
	3.9	4.4×10^{-6}	1225	0.775		
	4.1	4.3×10^{-6}	1322	0.770		
	4.4	3.9×10^{-6}	1420	0.791		
	5.0	4.4×10^{-6}	1555	0.829	1515	0.898
	5.3	3.4×10^{-6}	1289	0.784		
2	5.6	3.4×10^{-6}	1552	0.830	1515	0.894
	5.9	4.8×10^{-6}	1649	0.853	1630	0.885
	6.4	3.2×10^{-6}	1700	0.882	1680	0.915
	6.8	3.0×10^{-6}	1800	0.876	1781	0.906
	7.0	2.6×10^{-6}	1900	0.908	1883	0.934

TABLE 45

Total Hemispherical Emittance
Coating: Iron Titanate Plus Alumina (4-Mil)
Substrate: Columbium -1% Zirconium

Run Number	Elapsed Time (Hrs.)	Pressure (mm Hg)	Thermocouple Temp. (°F)	Thermocouple ϵ th	Optical Pyrometer Temp. (°F)	Optical Pyrometer ϵ th
1	0.7	3.7×10^{-6}	322	0.813		
	1.1	3.8×10^{-6}	524	0.827		
	1.6	4.0×10^{-6}	722	0.821		
	2.6	3.7×10^{-6}	921	0.803		
	3.0	3.8×10^{-6}	1021	0.783		
	3.3	4.0×10^{-6}	1121	0.761		
	3.6	4.4×10^{-6}	1222	0.740		
	3.8	4.5×10^{-6}	1321	0.708		
	4.1	4.3×10^{-6}	1424	0.730		
	4.7	3.5×10^{-6}	1521	0.781	1515	0.790
	5.3	4.5×10^{-6}	1621	0.782	1564	0.874
	5.8	3.4×10^{-6}	1721	0.897	1722	0.895
	6.0	3.0×10^{-6}	1826	0.893	1838	0.875
	6.2	2.6×10^{-6}	1920	0.895	1931	0.879
	6.4	2.6×10^{-6}	2019	0.926	2045	0.888
	6.6	3.0×10^{-6}	1621	0.888	1628	0.876
	6.8	2.5×10^{-6}	1023	0.825		

TABLE 46

Total Hemispherical Emittance
Coating: Strontium Titanate (10-Mil)
Substrate: AISI - 310 Stainless Steel

Run Number	Elapsed Time (Hrs)	Pressure (mm Hg)	Thermocouple Temp. (°F)	Thermocouple ϵ th	Optical Pyrometer Temp. (°F)	Optical Pyrometer ϵ th
1	0.4	6.1×10^{-6}	323	0.653		
	1.1	6.6×10^{-6}	526	0.652		
	1.4	6.3×10^{-6}	726	0.642		
	1.7	6.6×10^{-6}	928	0.558		
	2.1	6.4×10^{-6}	1029	0.518		
	2.4	6.8×10^{-6}	1126	0.456		
	2.6	8.1×10^{-6}	1225	0.426		
	2.8	6.8×10^{-6}	1323	0.406		
	3.0	7.6×10^{-6}	1423	0.394		
	3.1	8.3×10^{-6}	1525	0.379	1509	0.391
	3.3	7.5×10^{-6}	1272	0.405		
	3.4	7.3×10^{-6}	1071	0.443		

TABLE 47

Total Hemispherical Emittance
Coating: Strontium Titanate (3-Mil)
Substrate: Columbium -1% Zirconium

Run Number	Elapsed Time (Hrs.)	Pressure (mm Hg)	Thermocouple Temp. (°F)	Thermocouple ϵ th	Optical Pyrometer Temp. (°F)	Optical Pyrometer ϵ th
1	0.5	3.5×10^{-6}	323	0.826		
	1.3	5.0×10^{-6}	524	0.802		
	1.8	4.9×10^{-6}	724	0.788		
	2.0	4.8×10^{-6}	926	0.794		
	2.2	8.6×10^{-6}	1025	0.780		
	3.0	4.6×10^{-6}	1123	0.831		
	3.2	6.4×10^{-6}	1223	0.854		
	3.5	5.6×10^{-6}	1322	0.885		
	3.9	4.8×10^{-6}	1424	0.933		
	4.4	4.6×10^{-6}	1523	0.891	1496	0.941
	4.6	4.4×10^{-6}	1273	0.901		
	4.9	5.6×10^{-6}	1073	0.894		
2	5.3	4.5×10^{-6}	1524	0.893	1494	0.950
	5.5	4.2×10^{-6}	1622	0.905	1599	0.946
	5.8	4.0×10^{-6}	1171	0.693		
	6.1	7.8×10^{-8}	820	0.883		

TABLE 48

Total Hemispherical Emittance
Coating: Strontium Titanate (5-Mil)
Substrate: Columbium-1% Zirconium

Run Number	Elapsed Time (Hrs.)	Pressure (mm Hg)	Thermocouple Temp. (°F)	Thermocouple ϵ th	Optical Pyrometer Temp. (°F)	Optical Pyrometer ϵ th
1	0.7	1.5×10^{-5}	324	0.803		
	1.0	1.1×10^{-5}	526	0.831		
	2.5	1.0×10^{-5}	725	0.805		
	2.8	1.0×10^{-5}	929	0.802		
	3.0	1.2×10^{-5}	1029	0.804		
	3.2	1.0×10^{-5}	1128	0.818		
	3.4	1.0×10^{-5}	1179	0.924		
	3.9	9.8×10^{-6}	1329	0.887		
	4.3	9.8×10^{-6}	1428	0.882		
	4.5	7.8×10^{-6}	1530	0.904	1508	0.945
	4.7	6.9×10^{-6}	1278	0.893		
	5.0	7.2×10^{-6}	1076	0.878		
2	5.2	6.8×10^{-6}	1530	0.902	1508	0.943
	5.4	7.3×10^{-6}	1632	0.875	1617	0.904
	5.6	6.6×10^{-6}	1479	0.916		
	5.7	8.9×10^{-6}	1277	0.893		
	5.9	6.8×10^{-6}	1075	0.885		

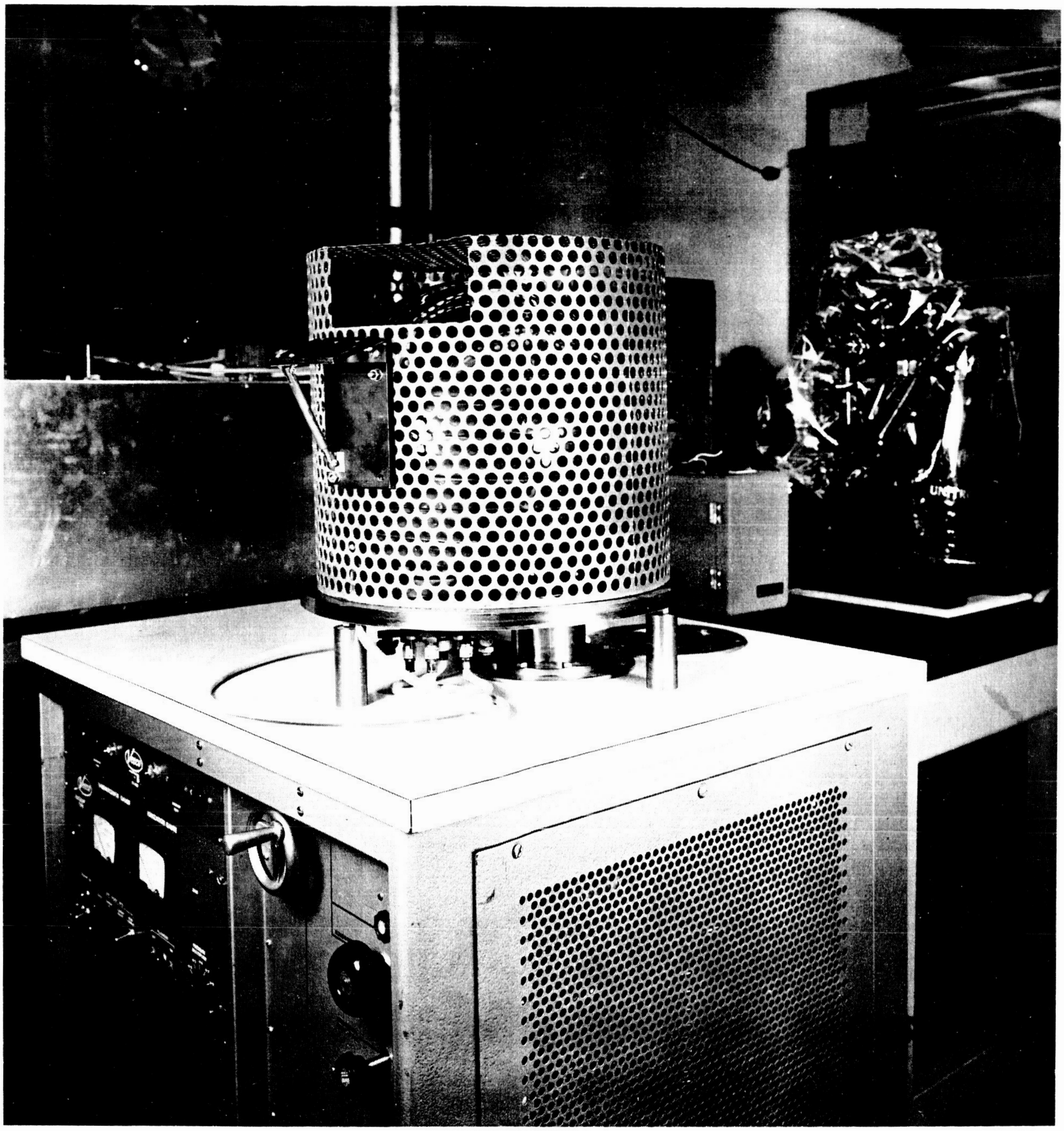
TABLE 49

Total Hemispherical Emittance
Coating: Silicon Carbide (5-Mil)
Substrate: Columbium -1% Zirconium

Run Number	Elapsed Time (Hrs.)	Pressure (mm Hg)	Thermocouple Temp. (°F)	Thermocouple ϵ_{th}	Optical Pyrometer Temp. (°F)	Optical Pyrometer ϵ_{th}
1	0.5	3.3×10^{-6}	330	0.851		
	2.0	4.2×10^{-6}	523	0.908		
	2.3	4.4×10^{-6}	723	0.923		
	2.6	7.0×10^{-6}	923	0.921		
	2.8	5.8×10^{-6}	1020	0.911		
	3.1	5.2×10^{-6}	1123	0.906		
	3.3	6.1×10^{-6}	1220	0.912		
	3.5	7.4×10^{-6}	1322	0.911		
	3.8	9.8×10^{-6}	1421	0.911		
	4.0	5.7×10^{-6}	1549	0.907	1523	0.956

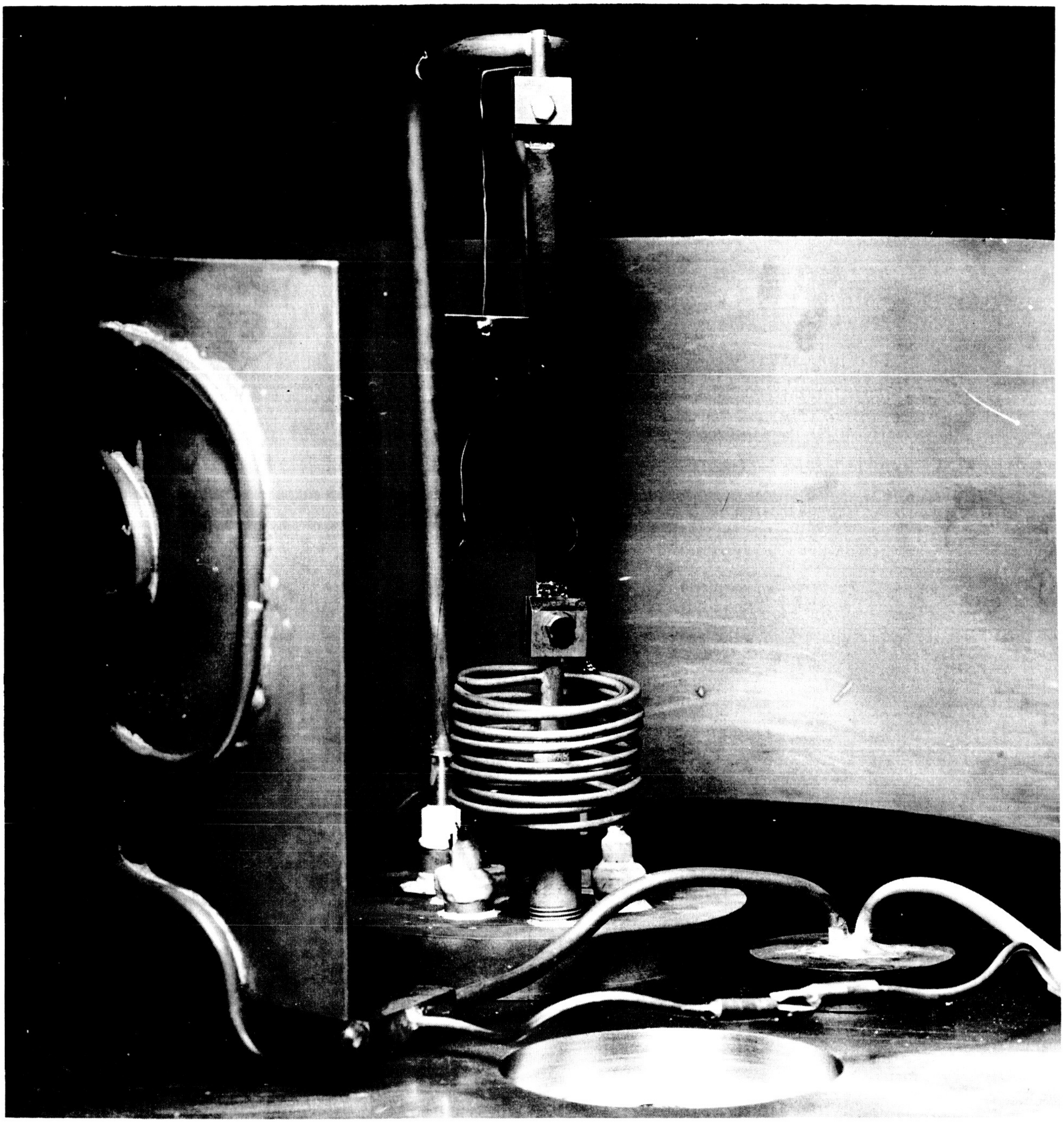
APPENDIX B

Figures



Volatilization Rig Mounted on Vacuum Station

Figure 1



Instrumentation Flange With Test Specimen
Installed in Volatilization Rig

Figure 2

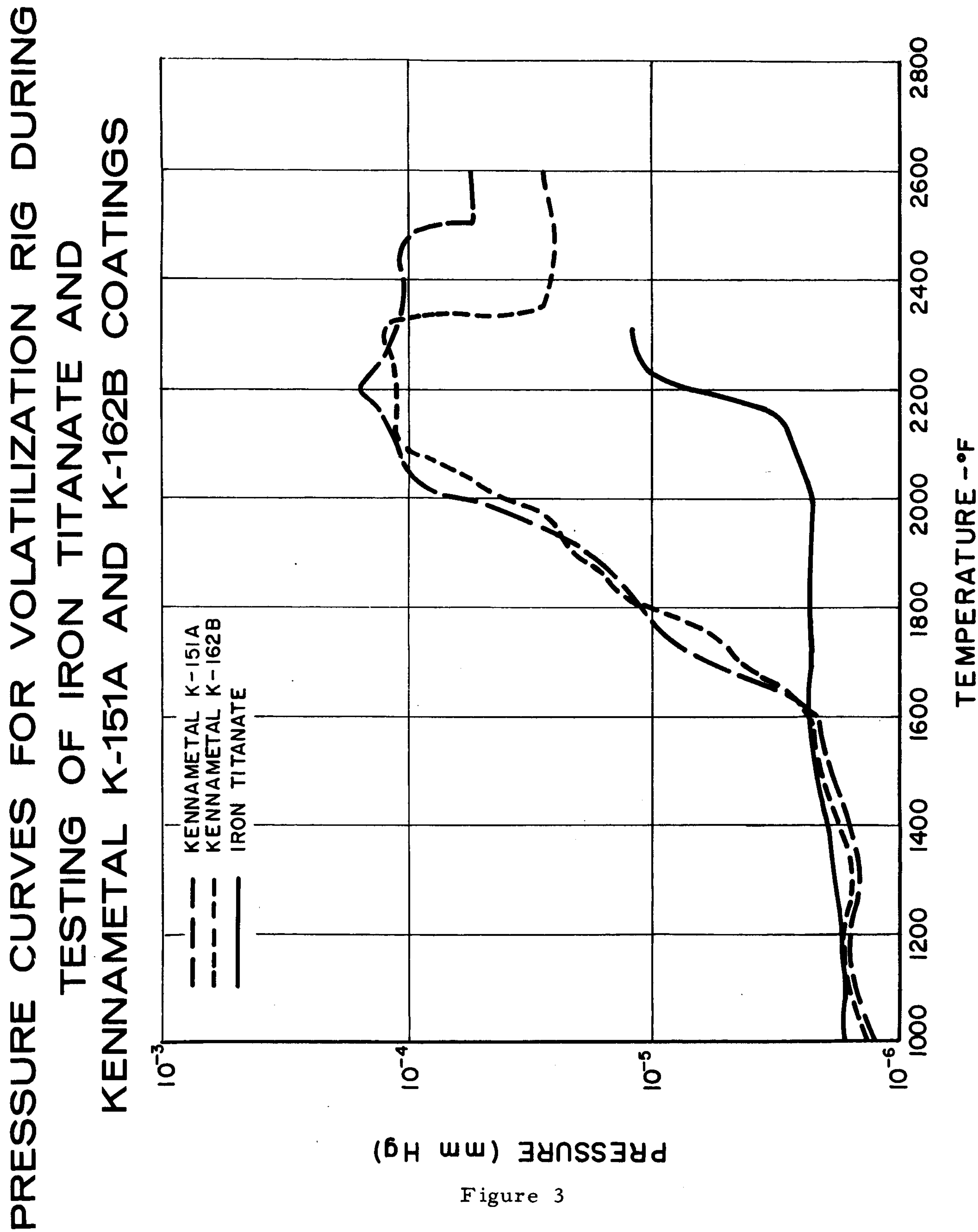

PRESSURE CURVES FOR VOLATILIZATION RIG DURING
TESTING OF IRON TITANATE AND
KENNAMETAL K-151A AND K-162B COATINGS
KENNAMETAL K-151A
KENNAMETAL K-162B
IRON TITANATE
PRESSURE (mm Hg)
10^{-3}
10^{-4}
10^{-5}
10^{-6}
TEMPERATURE - °F
1000
1200
1400
1600
1800
2000
2200
2400
2600
2800

Figure 3

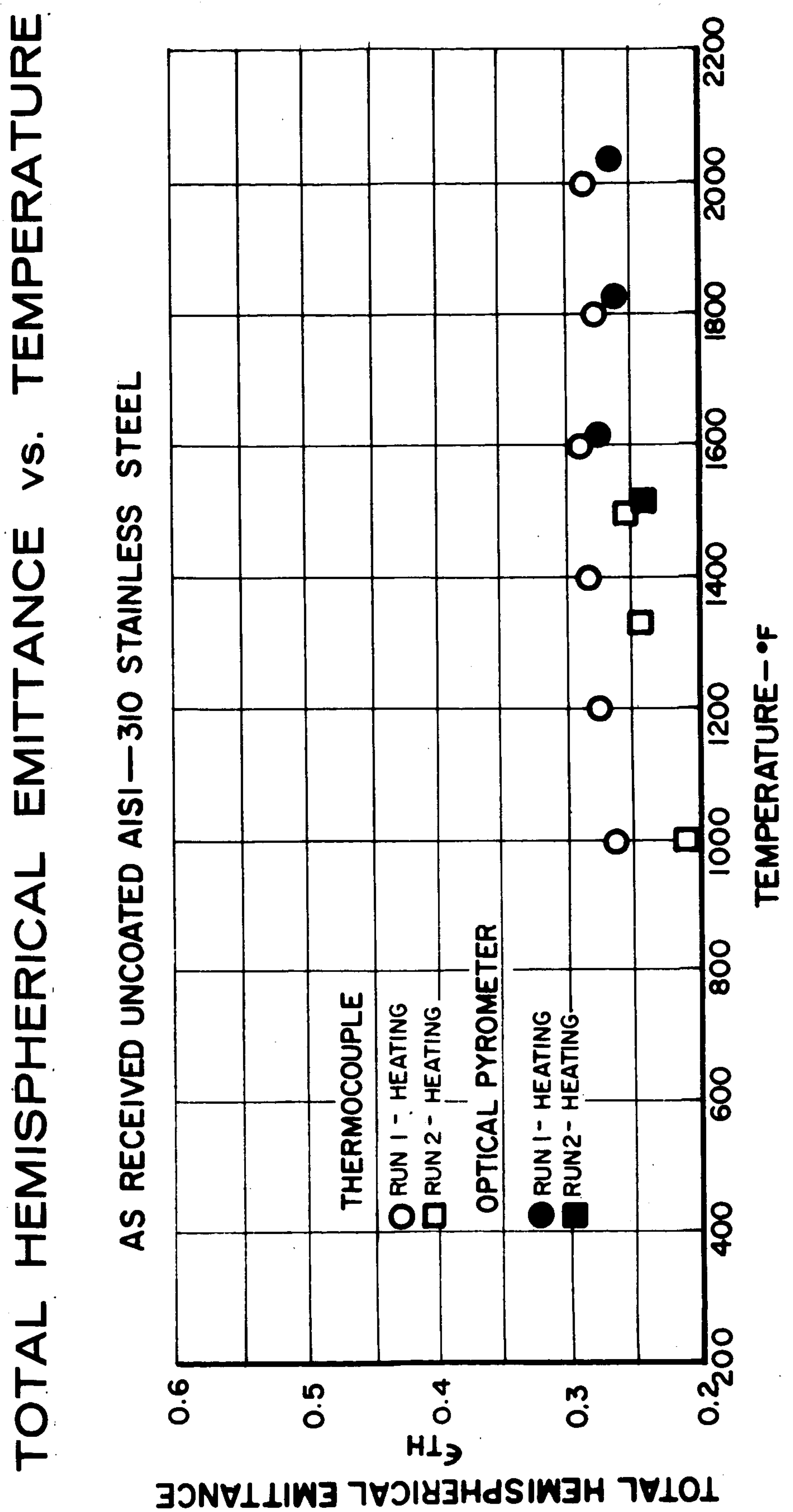

TOTAL HEMISPHERICAL EMITTANCE vs. TEMPERATURE
AS RECEIVED UNCOATED AISI — 310 STAINLESS STEEL
THERMOCOUPLE
RUN 1 - HEATING
RUN 2 - HEATING
OPTICAL PYROMETER
RUN 1 - HEATING
RUN 2 - HEATING
TEMPERATURE — °F
200
400
600
800
1000
1200
1400
1600
1800
2000
2200
TOTAL HEMISPHERICAL EMITTANCE
ϵ_{TH}
0.2
0.3
0.4
0.5
0.6

Figure 4

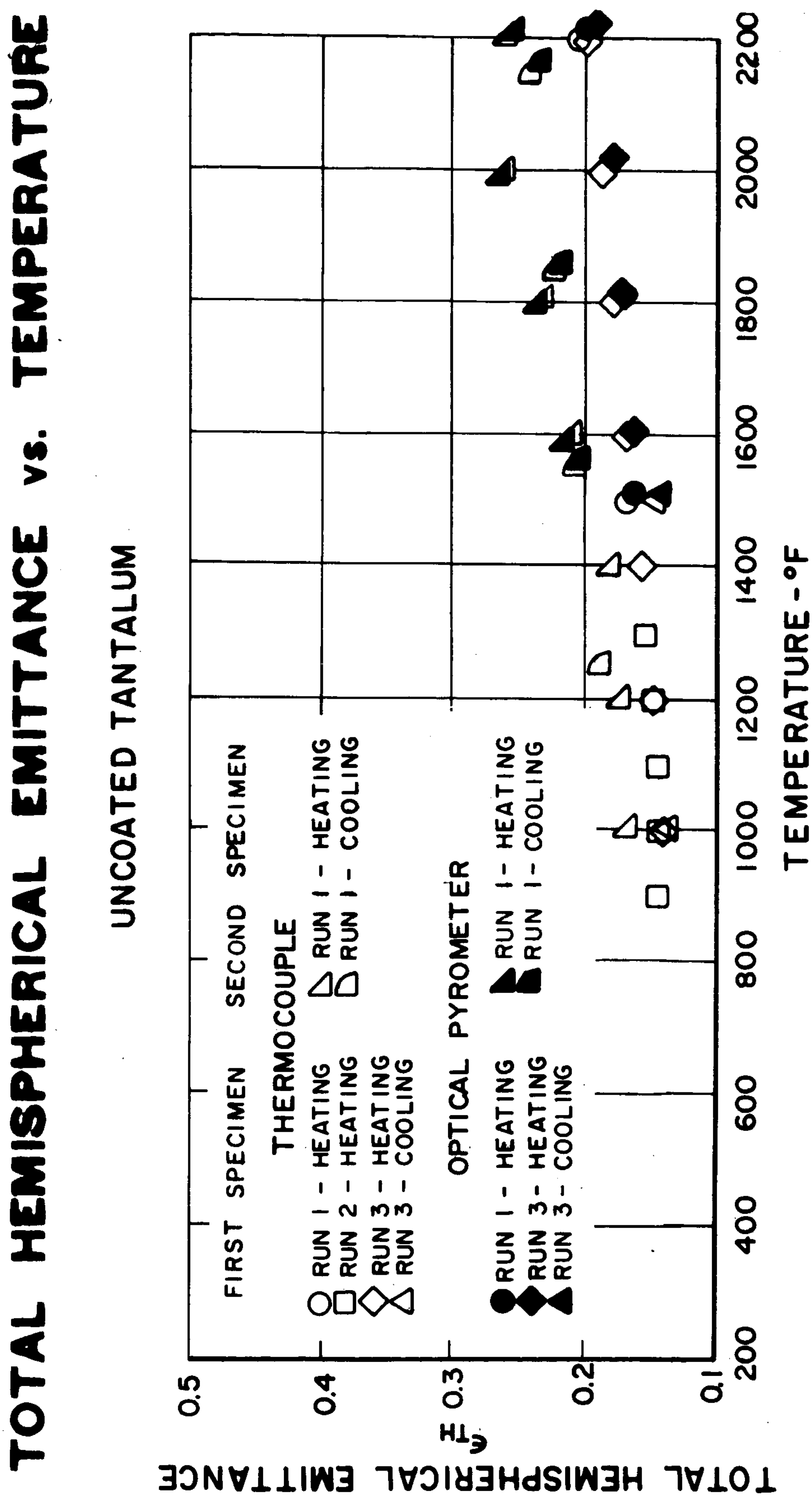

TOTAL HEMISPHERICAL EMITTANCE vs. TEMPERATURE
UNCOATED TANTALUM
FIRST SPECIMEN
SECOND SPECIMEN
THERMOCOUPLE
RUN 1 - HEATING
RUN 2 - HEATING
RUN 3 - HEATING
RUN 3 - COOLING
RUN 1 - HEATING
RUN 1 - COOLING
OPTICAL PYROMETER
RUN 1 - HEATING
RUN 3 - HEATING
RUN 3 - COOLING
RUN 1 - HEATING
RUN 1 - COOLING
0.5
0.4
0.3
0.2
0.1
ϵ_{TH}
TOTAL HEMISPHERICAL EMITTANCE
200
400
600
800
1000
1200
1400
1600
1800
2000
2200
TEMPERATURE - °F

Figure 5

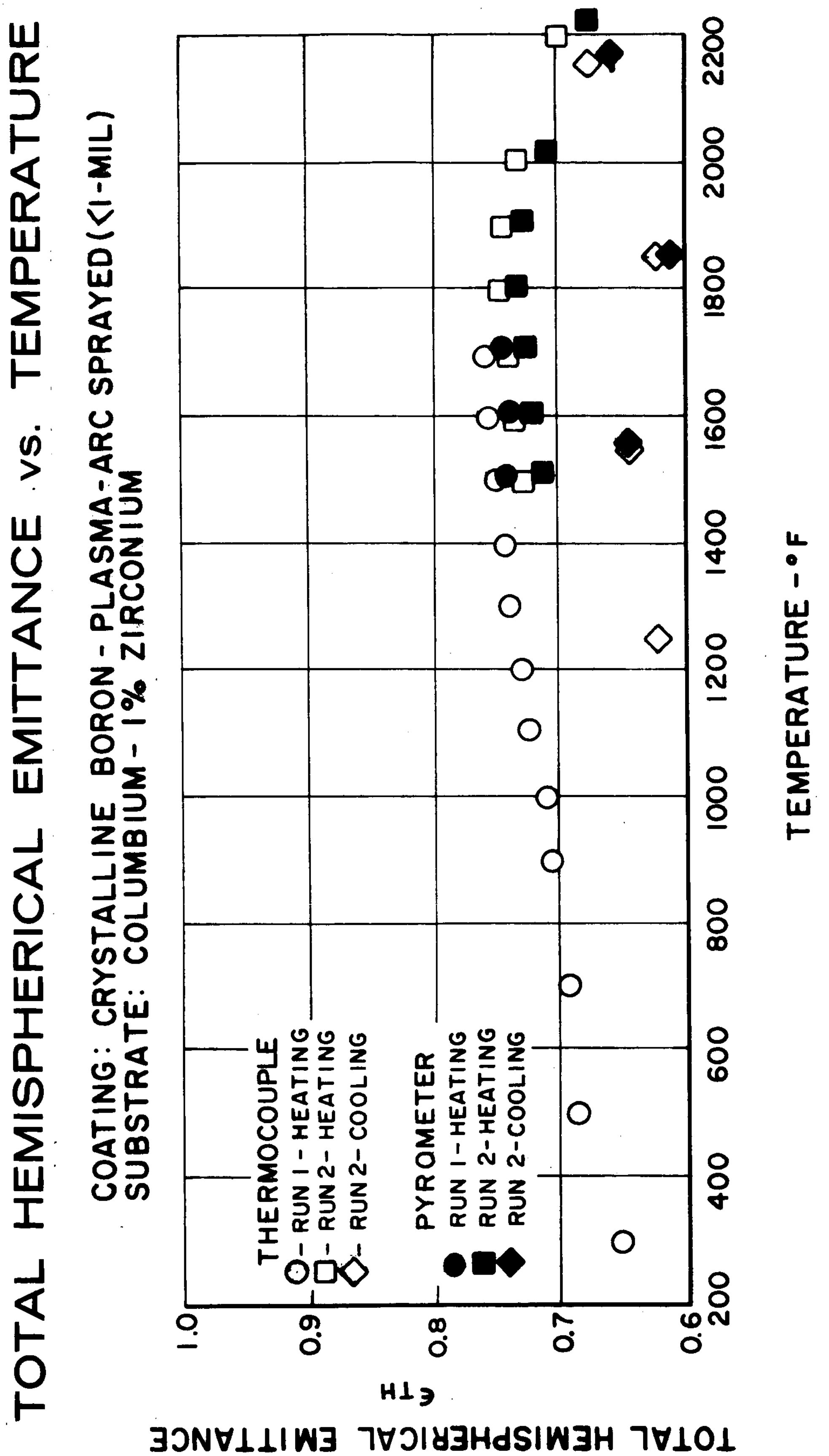

TOTAL HEMISPHERICAL EMITTANCE vs. TEMPERATURE
COATING: CRYSTALLINE BORON - PLASMA-ARC SPRAYED (<1-MIL)
SUBSTRATE: COLUMBIUM - 1% ZIRCONIUM
THERMOCOUPLE
- RUN 1 - HEATING
- RUN 2 - HEATING
- RUN 2 - COOLING
PYROMETER
RUN 1 - HEATING
RUN 2 - HEATING
RUN 2 - COOLING
1.0
0.9
0.8
0.7
0.6
200
400
600
800
1000
1200
1400
1600
1800
2000
2200
TEMPERATURE - °F
TOTAL HEMISPHERICAL EMITTANCE
ε TH

Figure 6

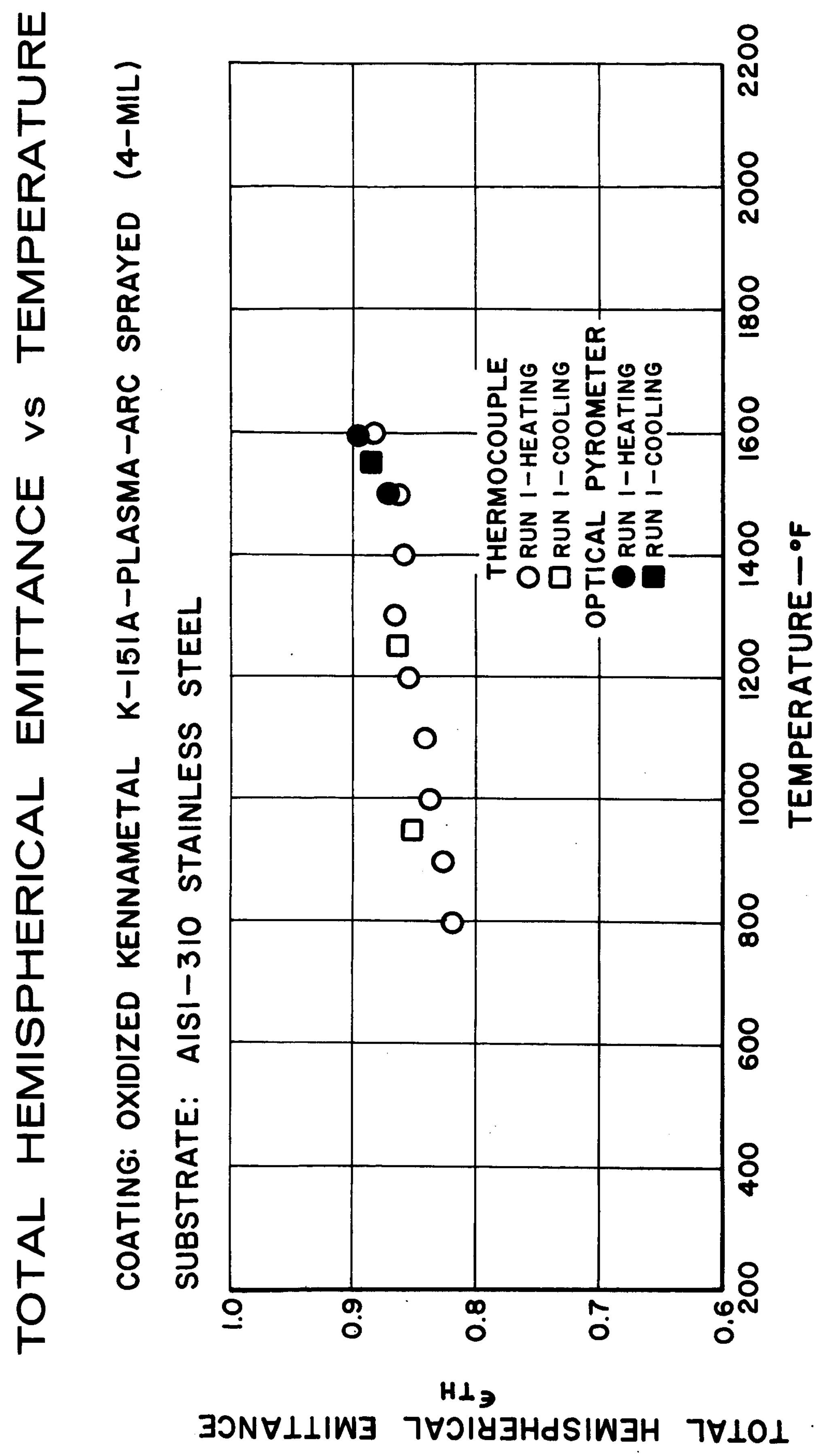

TOTAL HEMISPHERICAL EMITTANCE vs TEMPERATURE
COATING: OXIDIZED KENNAMETAL K-151A-PLASMA-ARC SPRAYED (4-MIL)
SUBSTRATE: AISI-310 STAINLESS STEEL
THERMOCOUPLE
RUN I-HEATING
RUN I-COOLING
OPTICAL PYROMETER
RUN I-HEATING
RUN I-COOLING
1.0
0.9
0.8
0.7
0.6
200
400
600
800
1000
1200
1400
1600
1800
2000
2200
TEMPERATURE—°F
TOTAL HEMISPHERICAL EMITTANCE
ϵ_{TH}

Figure 7

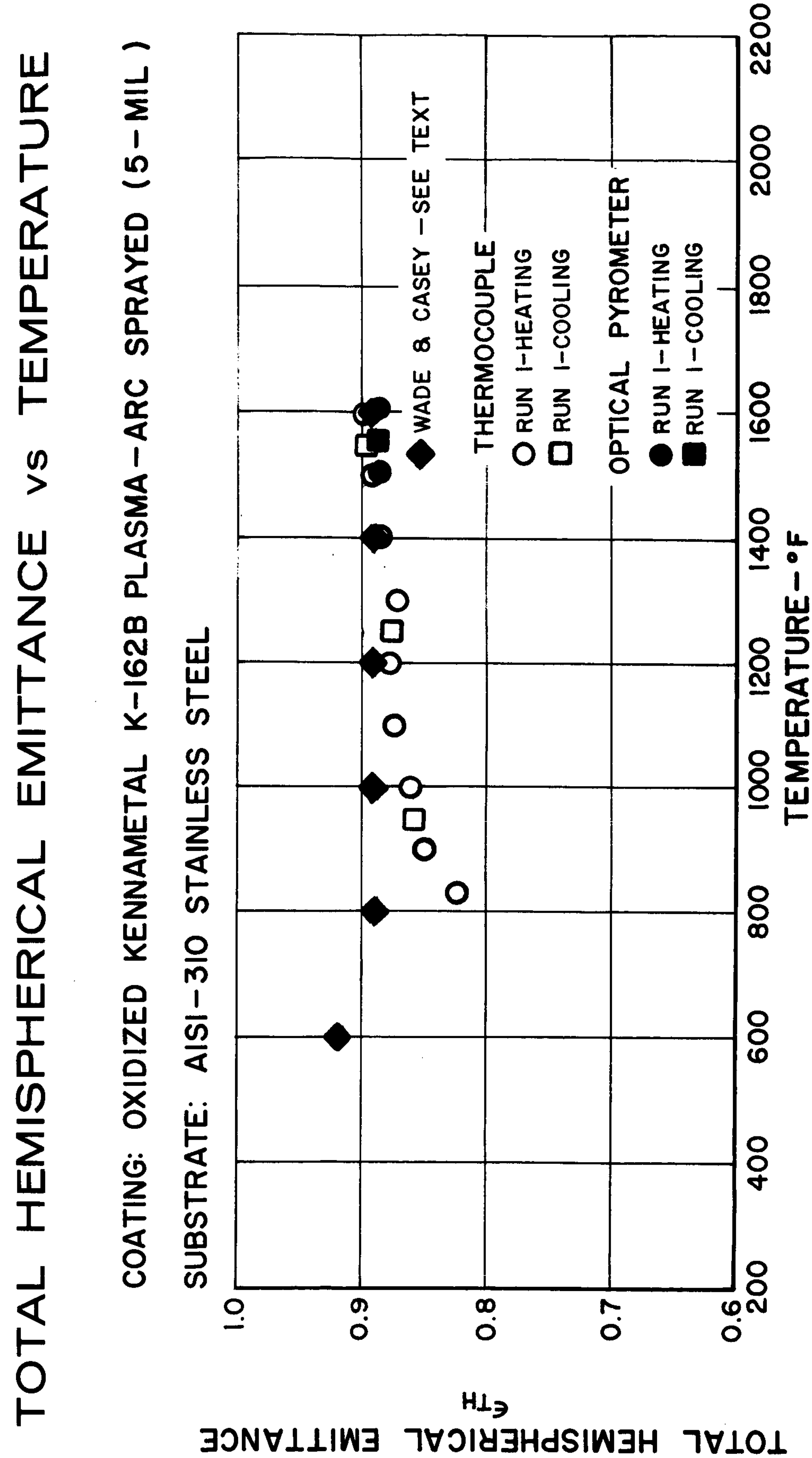

TOTAL HEMISPHERICAL EMITTANCE vs TEMPERATURE
COATING: OXIDIZED KENNAMETAL K-162B PLASMA-ARC SPRAYED (5-MIL)
SUBSTRATE: AISI-310 STAINLESS STEEL
WADE & CASEY - SEE TEXT
THERMOCOUPLE
RUN I-HEATING
RUN I-COOLING
OPTICAL PYROMETER
RUN I-HEATING
RUN I-COOLING
TOTAL HEMISPHERICAL EMITTANCE
ϵ_{TH}
1.0
0.9
0.8
0.7
0.6
200
400
600
800
1000
1200
1400
1600
1800
2000
2200
TEMPERATURE - °F

Figure 8

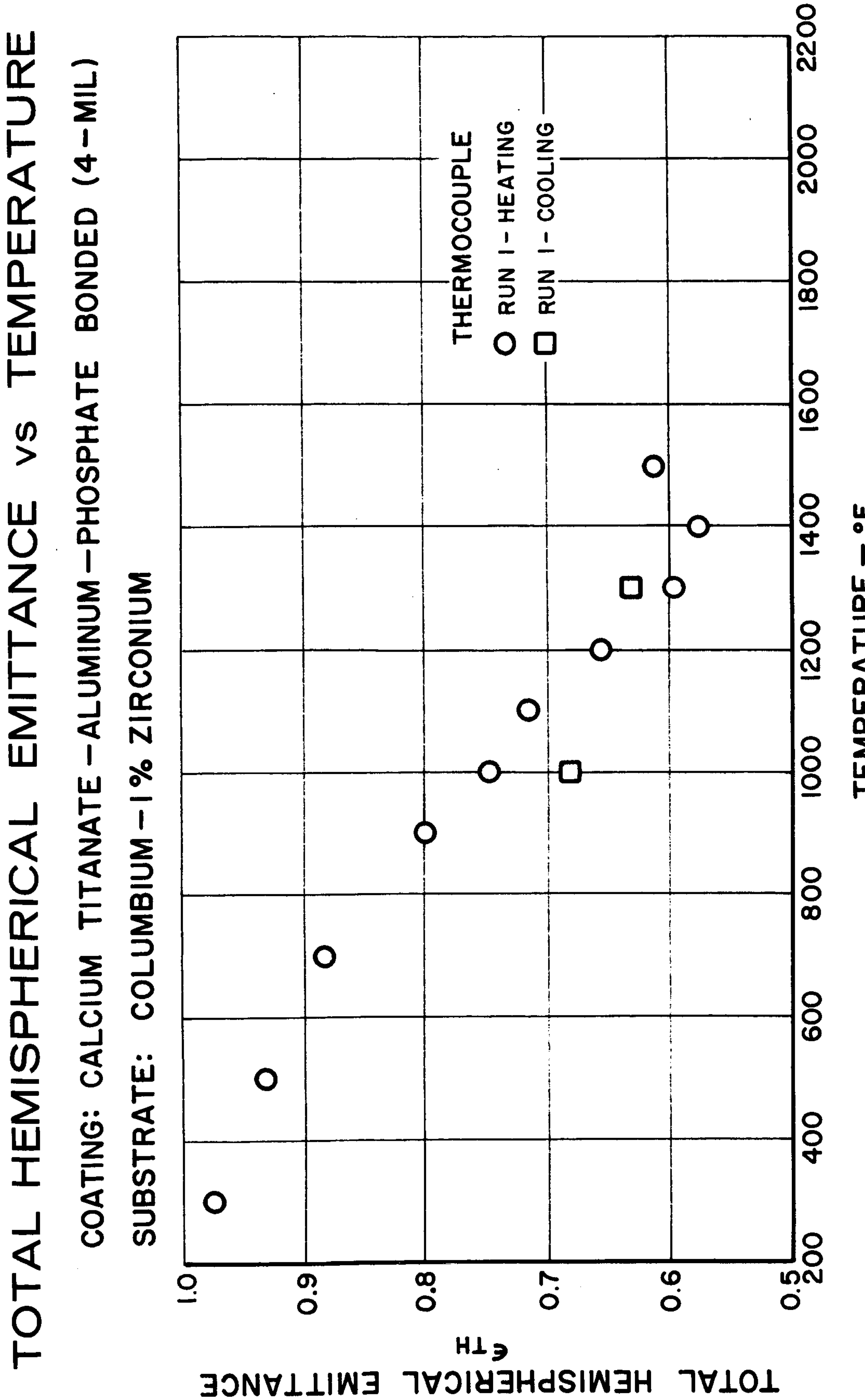

TOTAL HEMISPHERICAL EMITTANCE vs TEMPERATURE
COATING: CALCIUM TITANATE - ALUMINUM - PHOSPHATE BONDED (4-MIL)
SUBSTRATE: COLUMBIUM - 1% ZIRCONIUM
THERMOCOUPLE
RUN 1 - HEATING
RUN 1 - COOLING
TOTAL HEMISPHERICAL EMITTANCE
ϵ_{TH}
1.0
0.9
0.8
0.7
0.6
0.5
200
400
600
800
1000
1200
1400
1600
1800
2000
2200
TEMPERATURE — °F

Figure 9

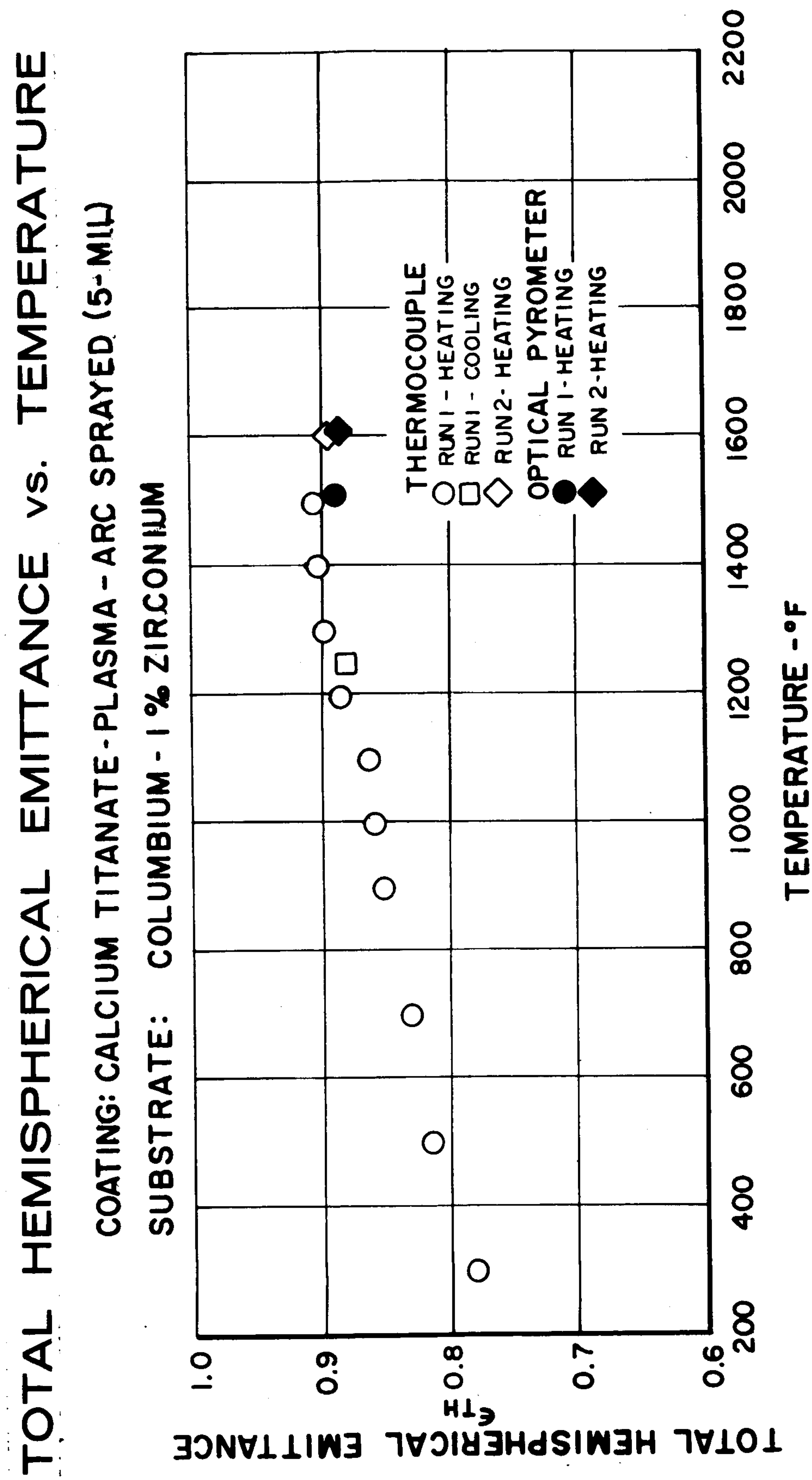

TOTAL HEMISPHERICAL EMITTANCE vs. TEMPERATURE
COATING: CALCIUM TITANATE - PLASMA - ARC SPRAYED (5-MIL)
SUBSTRATE: COLUMBIUM - 1 % ZIRCONIUM
THERMOCOUPLE
RUN 1 - HEATING
RUN 1 - COOLING
RUN 2 - HEATING
OPTICAL PYROMETER
RUN 1 - HEATING
RUN 2 - HEATING
TOTAL HEMISPHERICAL EMITTANCE
ε TH
1.0
0.9
0.8
0.7
0.6
200
400
600
800
1000
1200
1400
1600
1800
2000
2200
TEMPERATURE - °F

Figure 10

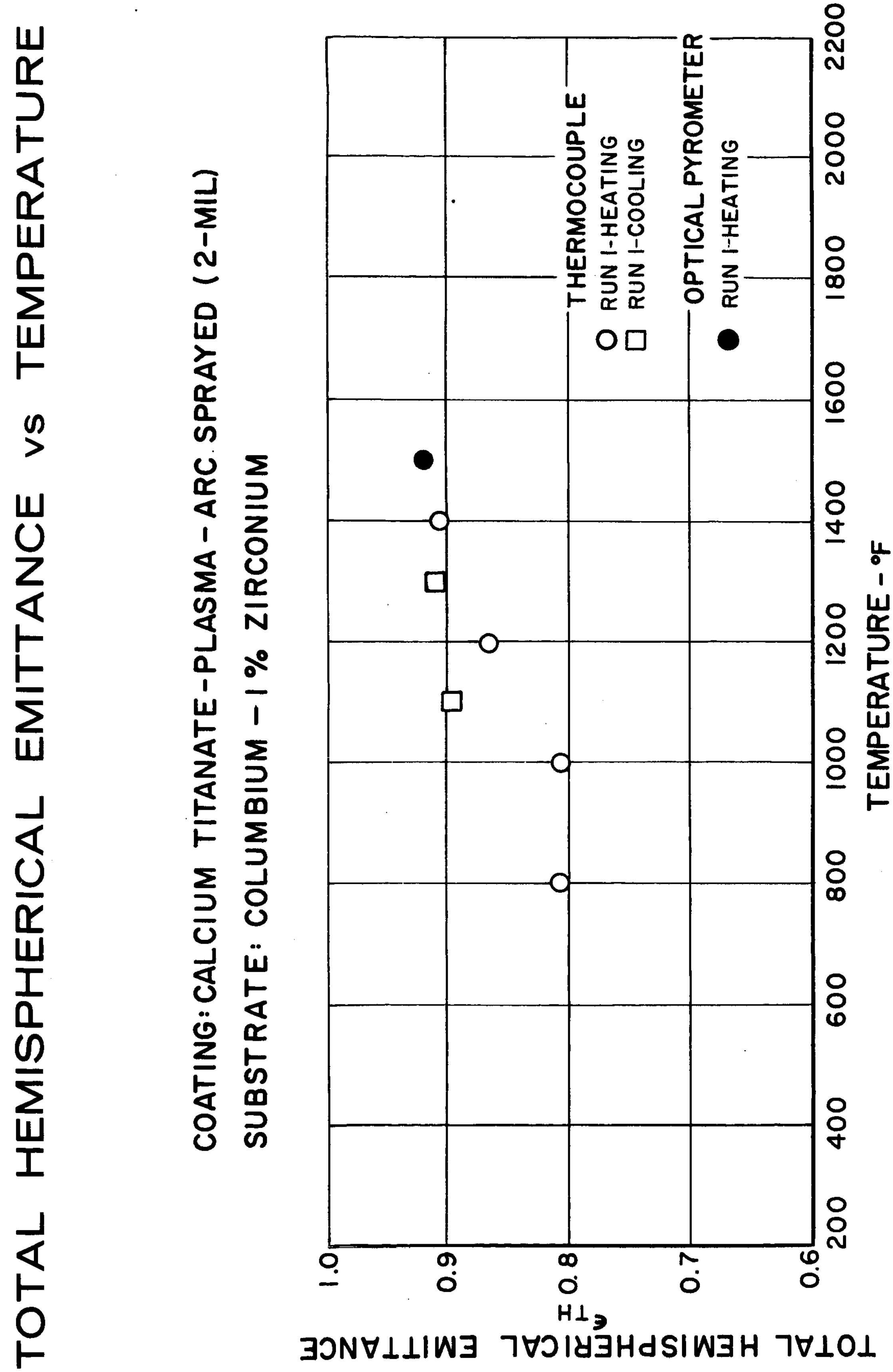

TOTAL HEMISPHERICAL EMITTANCE vs TEMPERATURE
COATING: CALCIUM TITANATE-PLASMA-ARC SPRAYED (2-MIL)
SUBSTRATE: COLUMBIUM-1% ZIRCONIUM
THERMOCOUPLE
RUN I-HEATING
RUN I-COOLING
OPTICAL PYROMETER
RUN I-HEATING
1.0
0.9
0.8
0.7
0.6
ϵ_{TH}
TOTAL HEMISPHERICAL EMITTANCE
200
400
600
800
1000
1200
1400
1600
1800
2000
2200
TEMPERATURE - °F

Figure 11

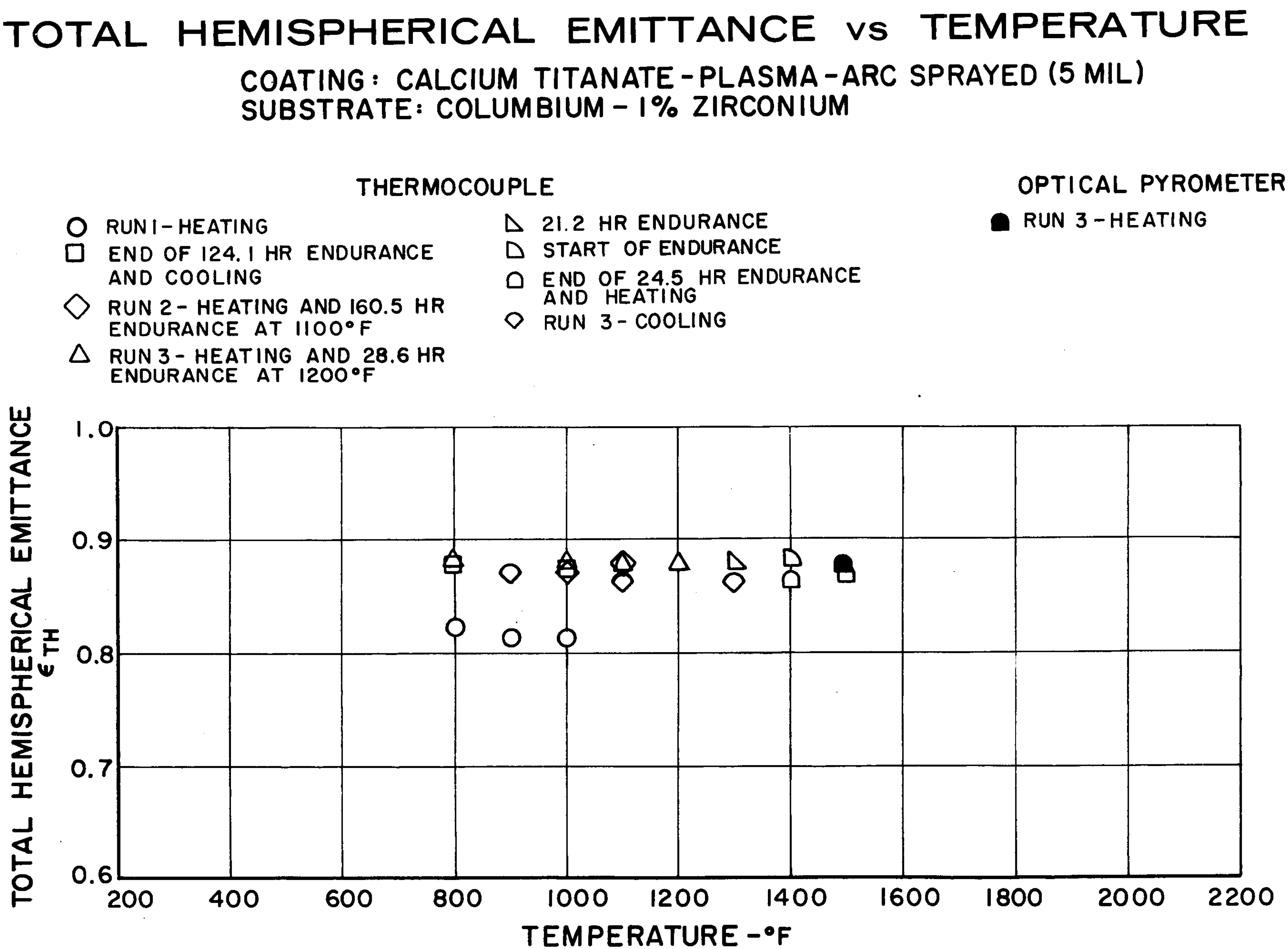

TOTAL HEMISPHERICAL EMITTANCE vs TEMPERATURE
COATING: CALCIUM TITANATE-PLASMA-ARC SPRAYED (5 MIL)
SUBSTRATE: COLUMBIUM - 1% ZIRCONIUM
THERMOCOUPLE
RUN 1 - HEATING
END OF 124.1 HR ENDURANCE AND COOLING
RUN 2 - HEATING AND 160.5 HR ENDURANCE AT 1100°F
RUN 3 - HEATING AND 28.6 HR ENDURANCE AT 1200°F
21.2 HR ENDURANCE
START OF ENDURANCE
END OF 24.5 HR ENDURANCE AND HEATING
RUN 3 - COOLING
OPTICAL PYROMETER
RUN 3 - HEATING
TOTAL HEMISPHERICAL EMITTANCE
ε TH
1.0
0.9
0.8
0.7
0.6
200
400
600
800
1000
1200
1400
1600
1800
2000
2200
TEMPERATURE - °F

Figure 12

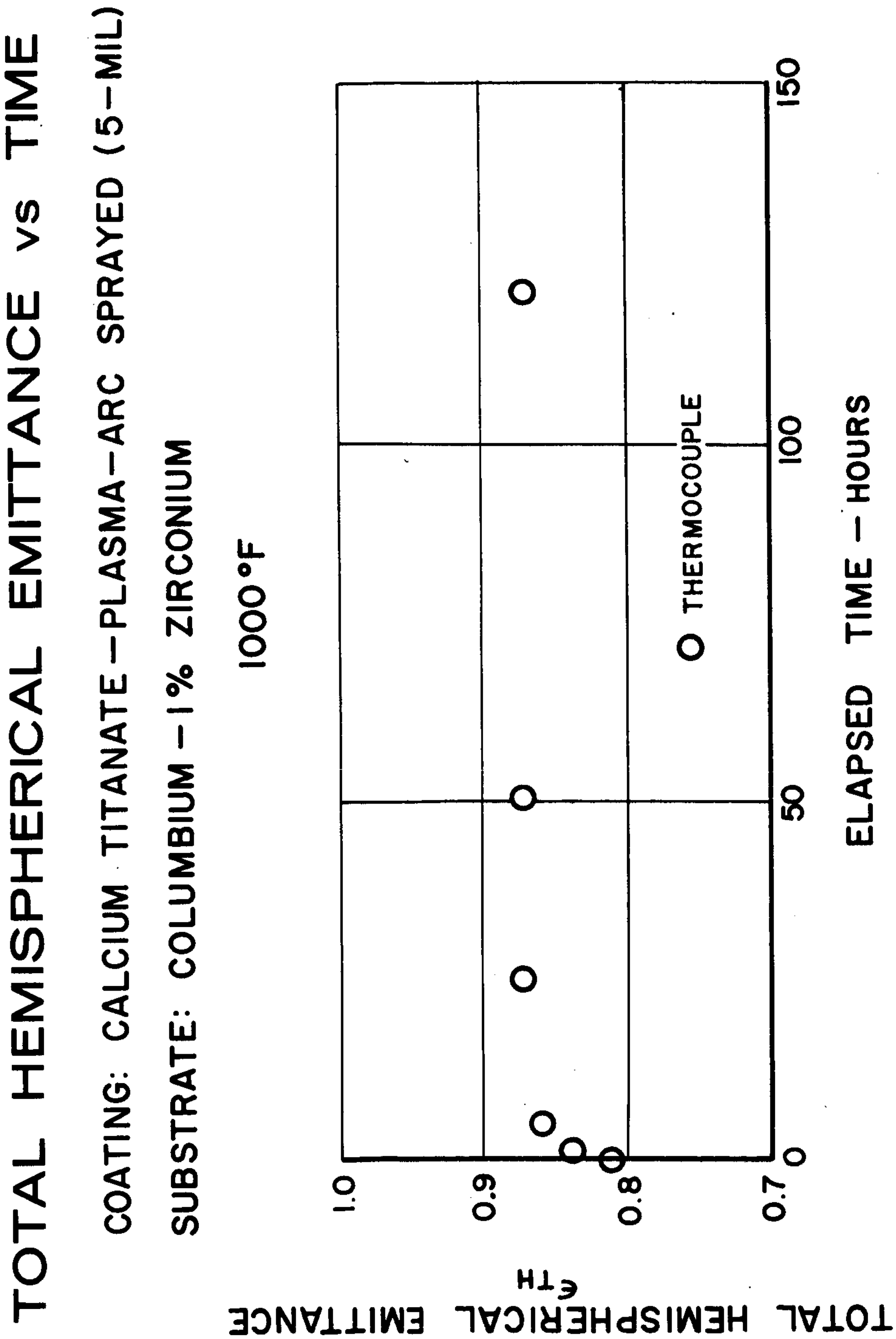

TOTAL HEMISPHERICAL EMITTANCE vs TIME
COATING: CALCIUM TITANATE—PLASMA—ARC SPRAYED (5—MIL)
SUBSTRATE: COLUMBIUM—1% ZIRCONIUM
1000°F
O THERMOCOUPLE
TOTAL HEMISPHERICAL EMITTANCE
ϵTH
1.0
0.9
0.8
0.7
0
50
100
150
ELAPSED TIME — HOURS

Figure 13

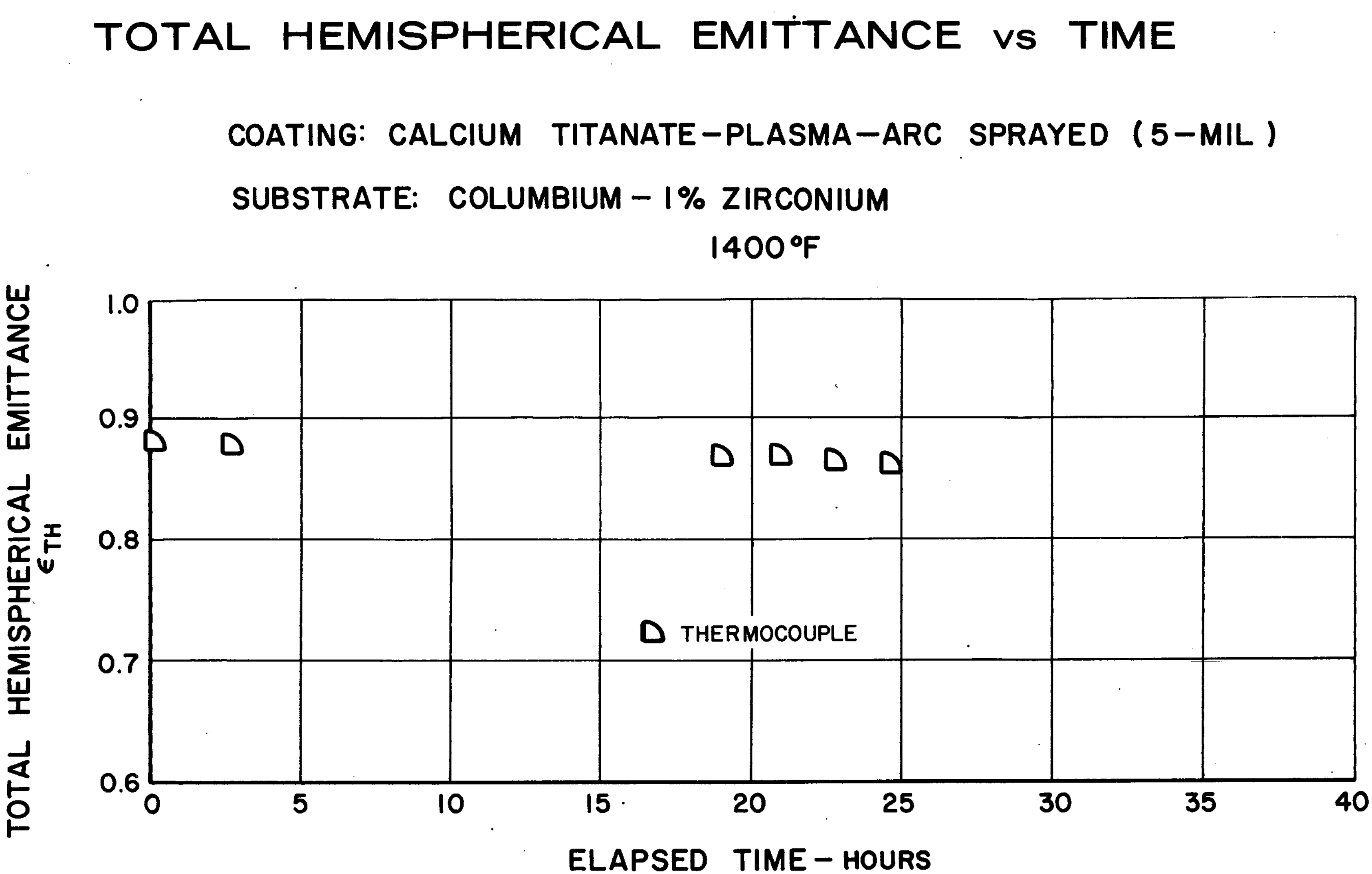

TOTAL HEMISPHERICAL EMITTANCE vs TIME
COATING: CALCIUM TITANATE-PLASMA-ARC SPRAYED (5-MIL)
SUBSTRATE: COLUMBIUM - 1% ZIRCONIUM
1400°F
TOTAL HEMISPHERICAL EMITTANCE ϵ_{TH}
1.0
0.9
0.8
0.7
0.6
0
5
10
15
20
25
30
35
40
ELAPSED TIME - HOURS
THERMOCOUPLE

Figure 14

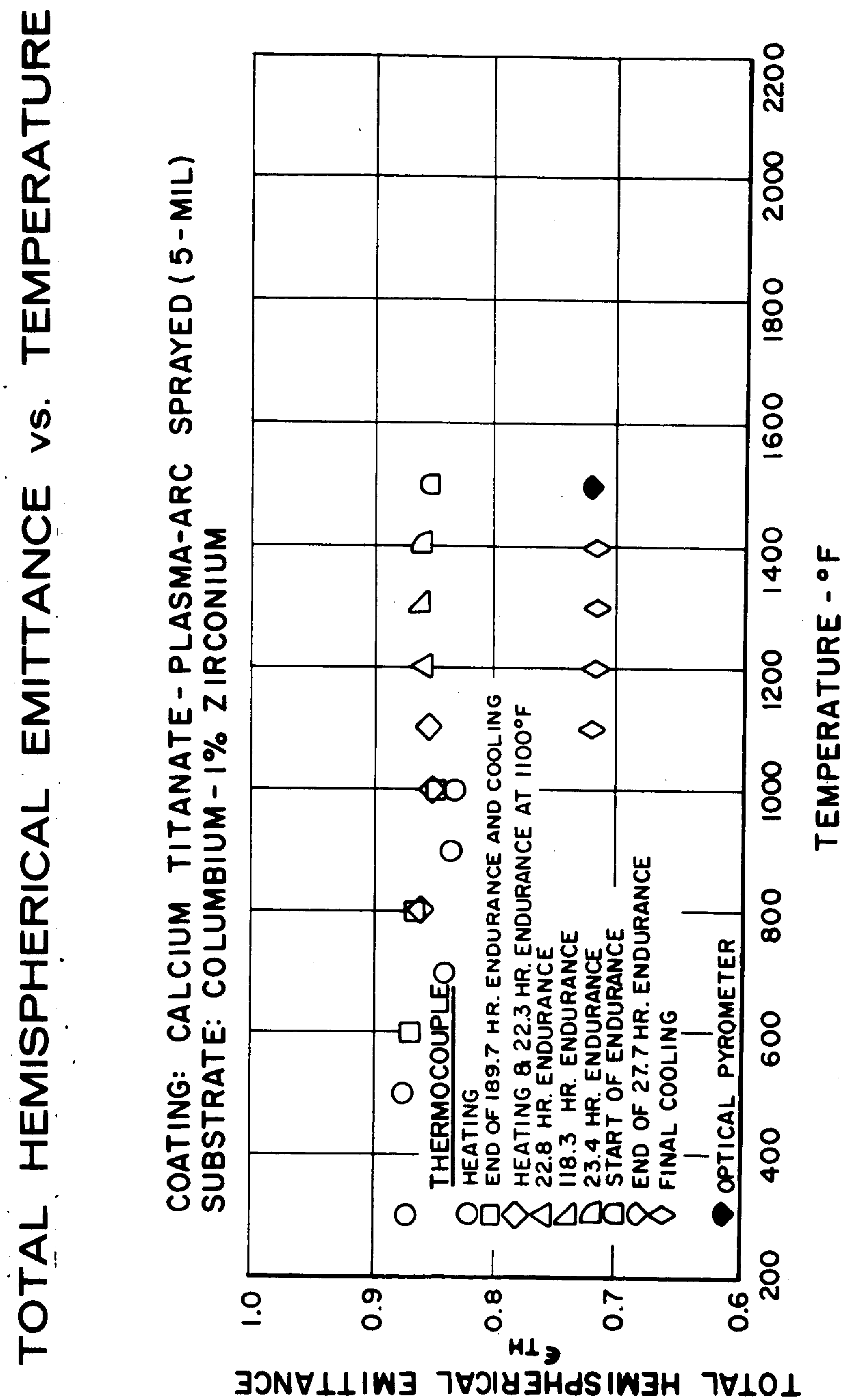

TOTAL HEMISPHERICAL EMITTANCE vs. TEMPERATURE
COATING: CALCIUM TITANATE - PLASMA-ARC SPRAYED (5-MIL)
SUBSTRATE: COLUMBIUM - 1% ZIRCONIUM
THERMOCOUPLE
HEATING
END OF 189.7 HR. ENDURANCE AND COOLING
HEATING & 22.3 HR. ENDURANCE AT 1100°F
22.8 HR. ENDURANCE
118.3 HR. ENDURANCE
23.4 HR. ENDURANCE
START OF ENDURANCE
END OF 27.7 HR. ENDURANCE
FINAL COOLING
OPTICAL PYROMETER
TOTAL HEMISPHERICAL EMITTANCE
ϵ_{TH}
1.0
0.9
0.8
0.7
0.6
200
400
600
800
1000
1200
1400
1600
1800
2000
2200
TEMPERATURE - °F

Figure 15

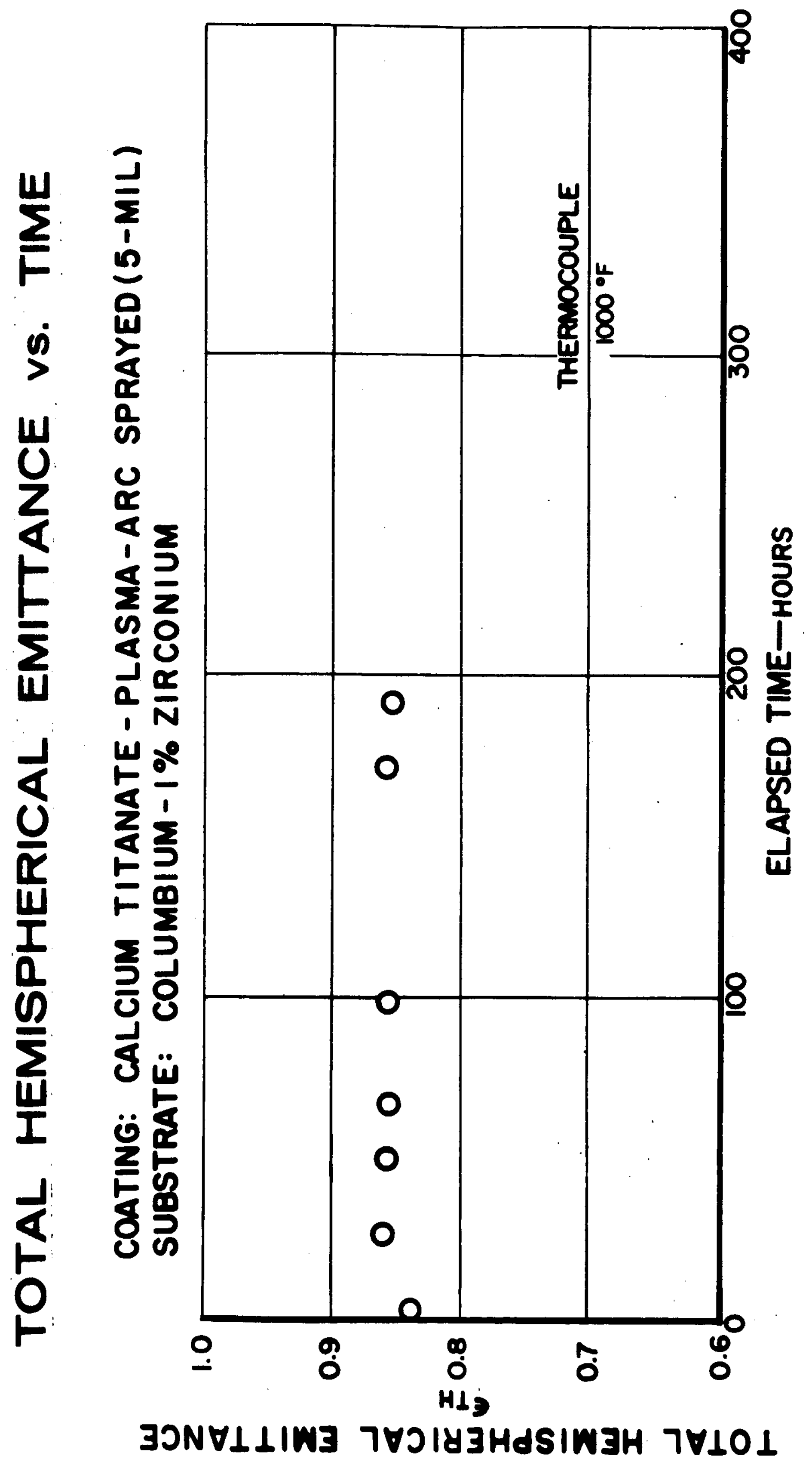

TOTAL HEMISPHERICAL EMITTANCE vs. TIME
COATING: CALCIUM TITANATE - PLASMA - ARC SPRAYED (5-MIL)
SUBSTRATE: COLUMBIUM - 1% ZIRCONIUM
THERMOCOUPLE
1000 °F
ELAPSED TIME—HOURS
0
100
200
300
400
1.0
0.9
0.8
0.7
0.6
ϵ_{TH}
TOTAL HEMISPHERICAL EMITTANCE

Figure 16

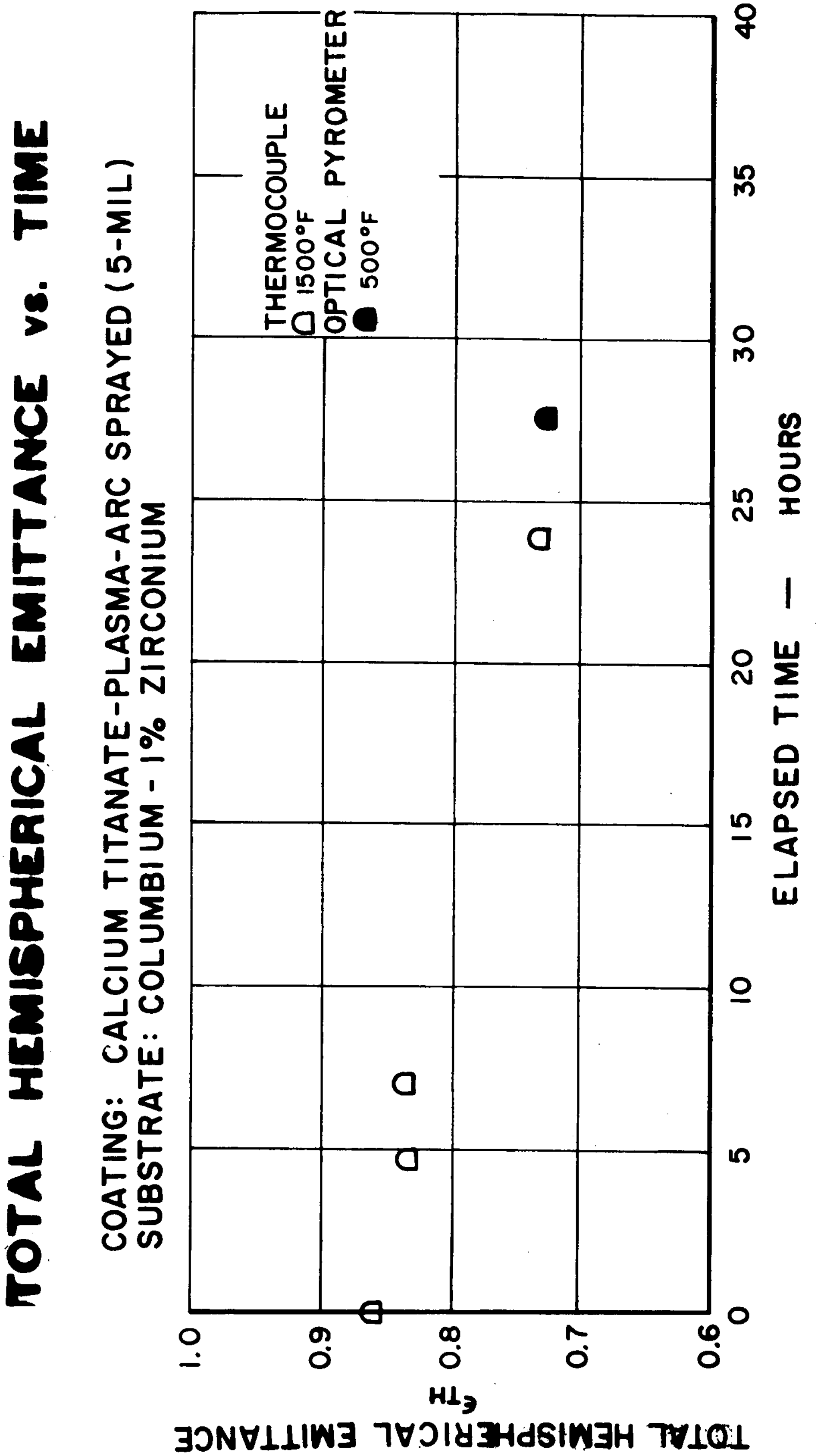

TOTAL HEMISPHERICAL EMITTANCE vs. TIME
COATING: CALCIUM TITANATE-PLASMA-ARC SPRAYED (5-MIL)
SUBSTRATE: COLUMBIUM - 1% ZIRCONIUM
THERMOCOUPLE
1500°F
OPTICAL PYROMETER
500°F
ELAPSED TIME — HOURS
0
5
10
15
20
25
30
35
40
1.0
0.9
0.8
0.7
0.6
TOTAL HEMISPHERICAL EMITTANCE
ϵ_{TH}

Figure 17

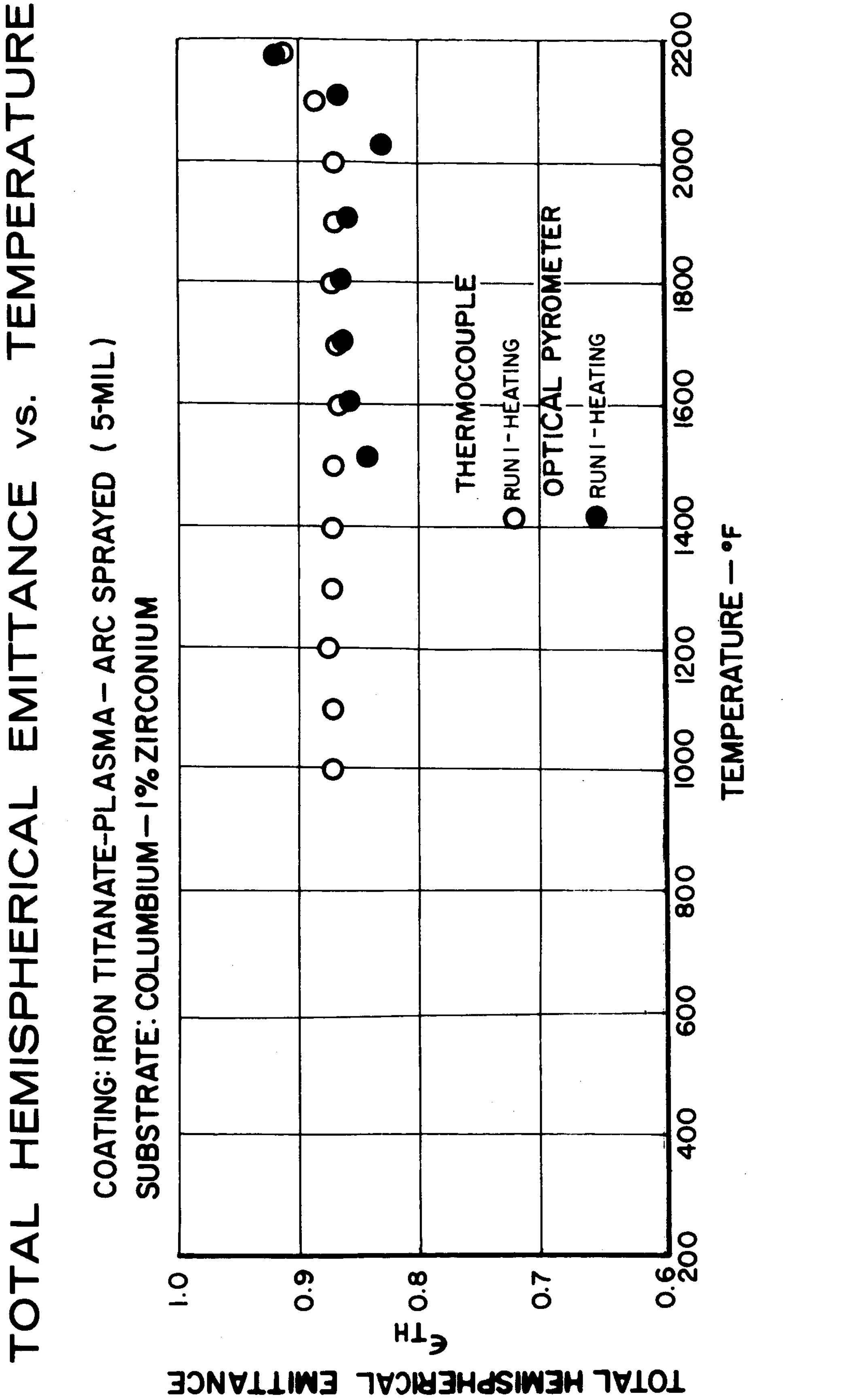

TOTAL HEMISPHERICAL EMITTANCE vs. TEMPERATURE
COATING: IRON TITANATE-PLASMA-ARC SPRAYED (5-MIL)
SUBSTRATE: COLUMBIUM-1% ZIRCONIUM
THERMOCOUPLE
RUN I-HEATING
OPTICAL PYROMETER
RUN I-HEATING
TOTAL HEMISPHERICAL EMITTANCE
ε TH
1.0
0.9
0.8
0.7
0.6
200
400
600
800
1000
1200
1400
1600
1800
2000
2200
TEMPERATURE — °F

Figure 18

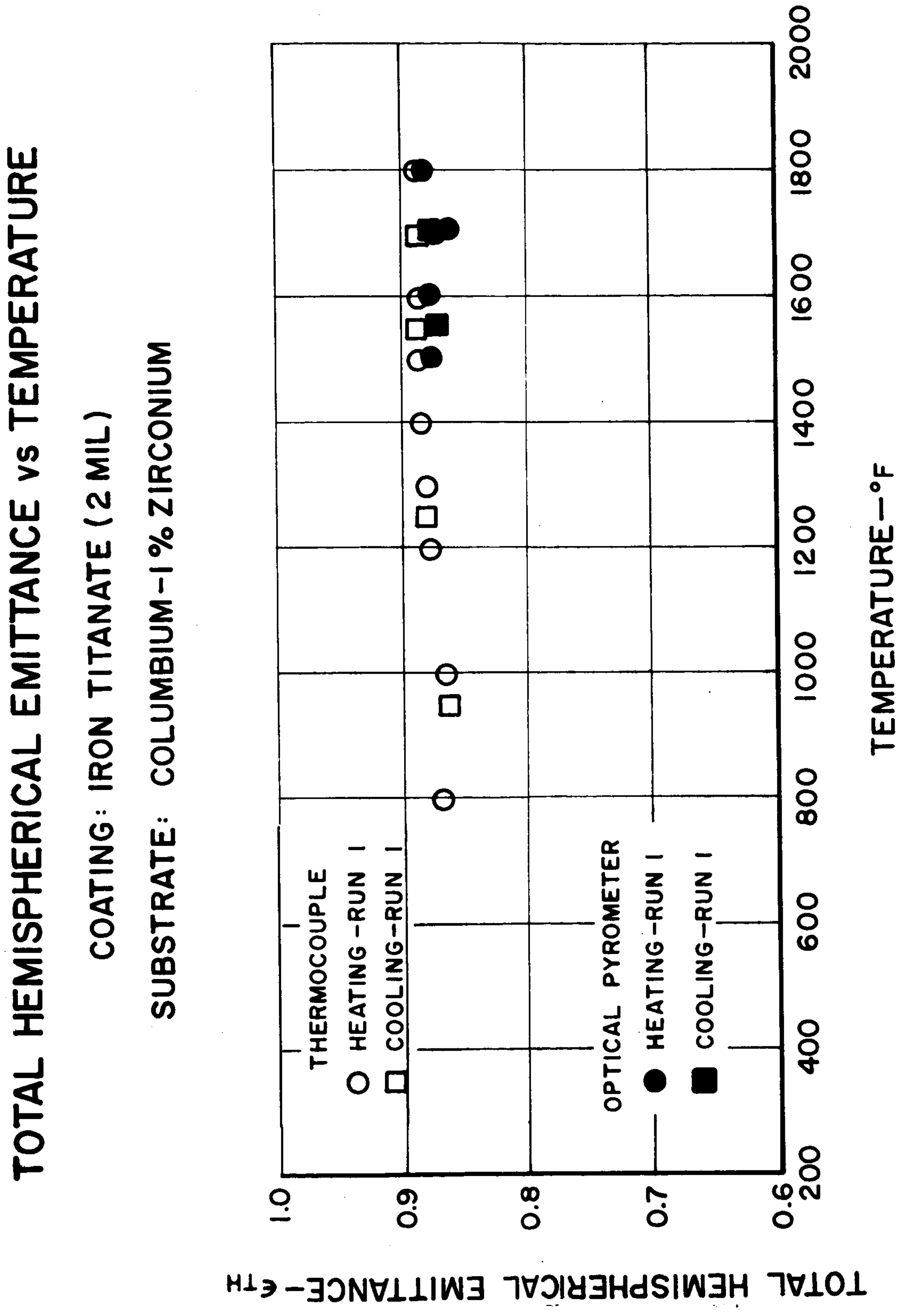

TOTAL HEMISPHERICAL EMITTANCE vs TEMPERATURE
COATING: IRON TITANATE (2 MIL)
SUBSTRATE: COLUMBIUM-1% ZIRCONIUM
THERMOCOUPLE
HEATING-RUN I
COOLING-RUN I
OPTICAL PYROMETER
HEATING-RUN I
COOLING-RUN I
1.0
0.9
0.8
0.7
0.6
200
400
600
800
1000
1200
1400
1600
1800
2000
TEMPERATURE—°F
TOTAL HEMISPHERICAL EMITTANCE—ϵ_{TH}

Figure 19

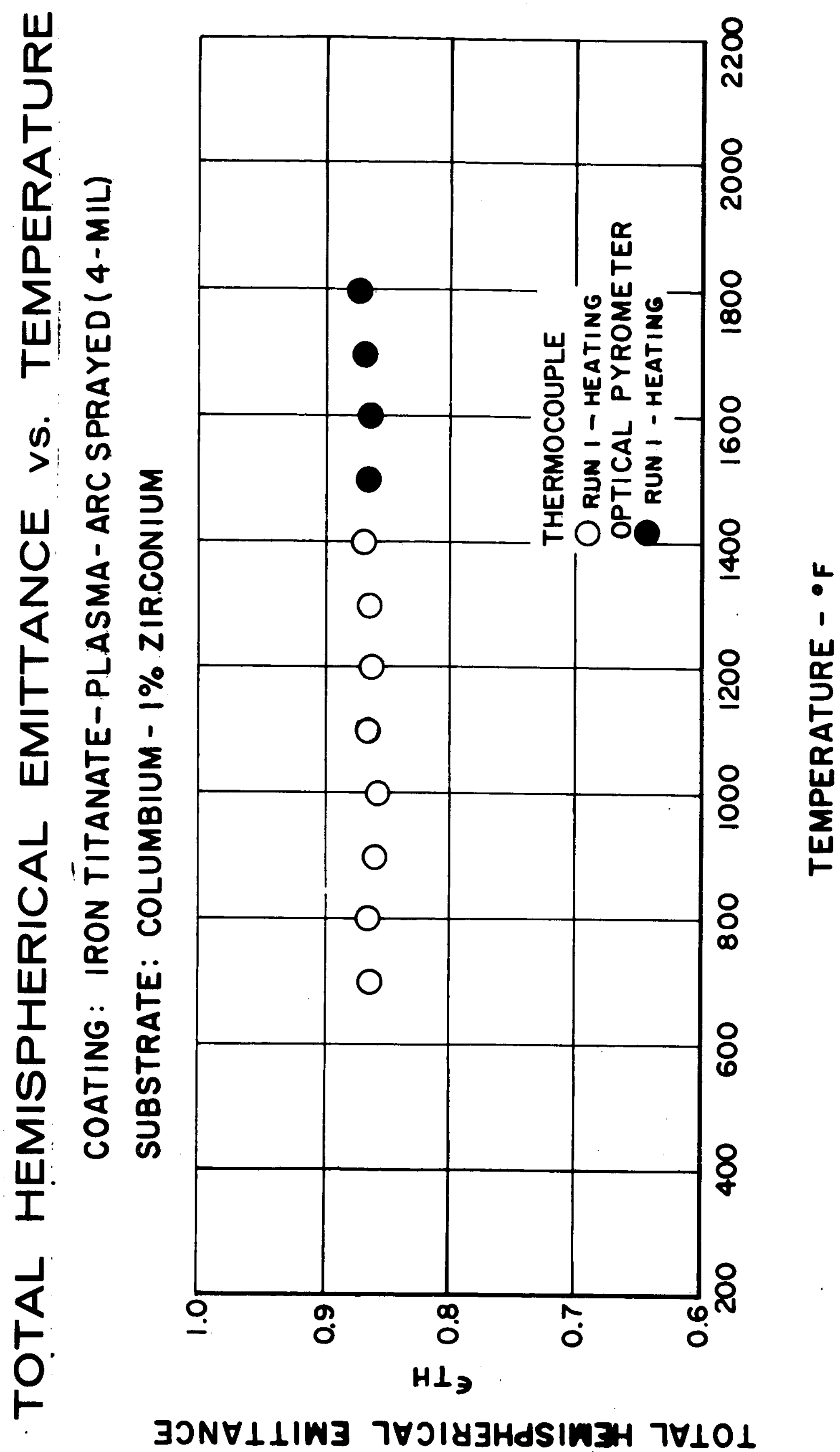
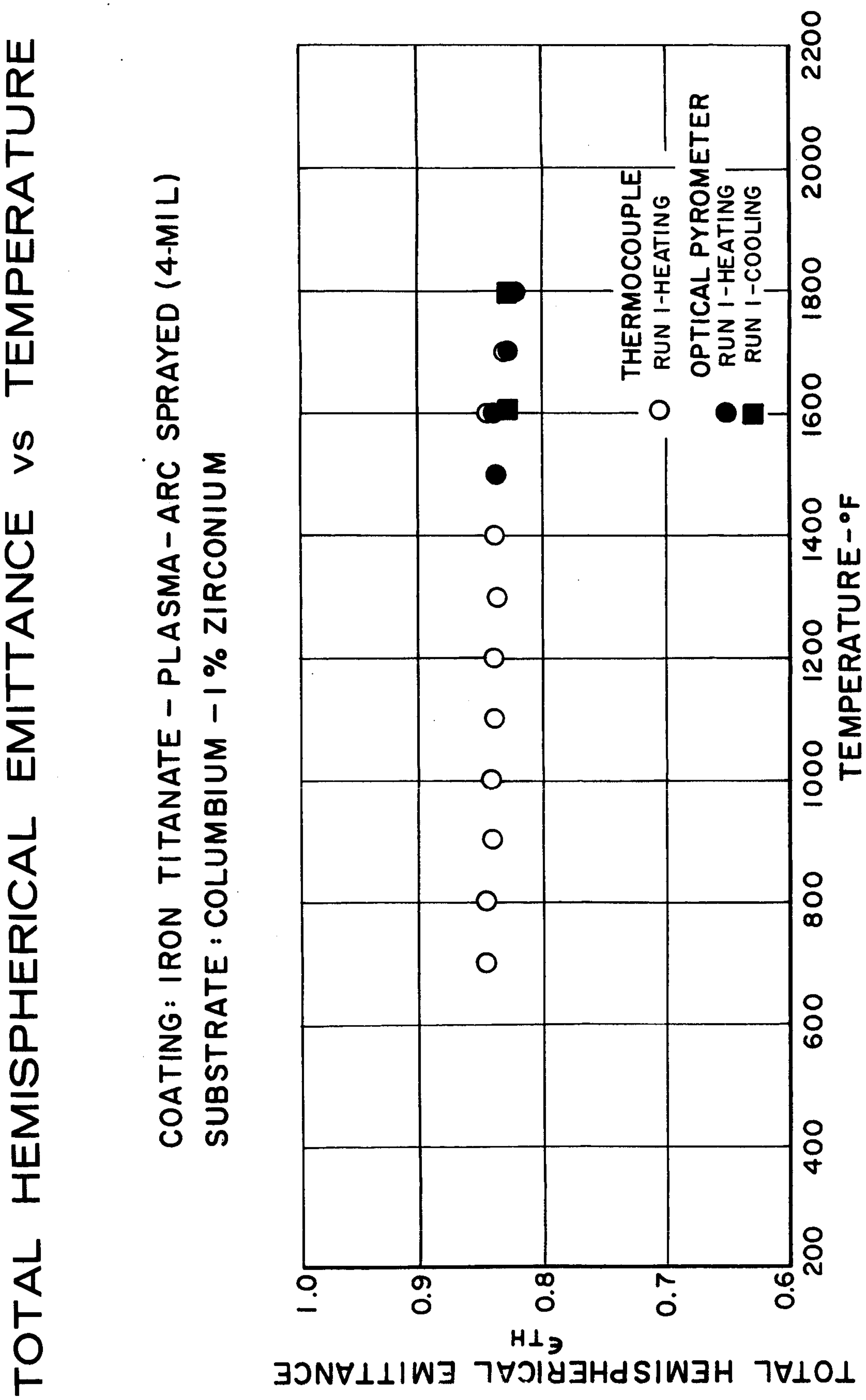

TOTAL HEMISPHERICAL EMITTANCE vs. TEMPERATURE
COATING: IRON TITANATE-PLASMA-ARC SPRAYED (4-MIL)
SUBSTRATE: COLUMBIUM - 1% ZIRCONIUM
THERMOCOUPLE
RUN I - HEATING
OPTICAL PYROMETER
RUN I - HEATING
1.0
0.9
0.8
0.7
0.6
200
400
600
800
1000
1200
1400
1600
1800
2000
2200
TEMPERATURE - °F
εTH
TOTAL HEMISPHERICAL EMITTANCE

Figure 20



TOTAL HEMISPHERICAL EMITTANCE vs TEMPERATURE
COATING: IRON TITANATE - PLASMA-ARC SPRAYED (4-MIL)
SUBSTRATE: COLUMBIUM - 1% ZIRCONIUM
THERMOCOUPLE
RUN 1-HEATING
OPTICAL PYROMETER
RUN 1-HEATING
RUN 1-COOLING
TOTAL HEMISPHERICAL EMITTANCE
ϵ_{TH}
1.0
0.9
0.8
0.7
0.6
200
400
600
800
1000
1200
1400
1600
1800
2000
2200
TEMPERATURE-°F

Figure 21

TOTAL HEMISPHERICAL EMITTANCE vs TIME

COATING: IRON TITANATE—PLASMA—ARC SPRAYED (4-MIL)
SUBSTRATE: COLUMBIUM—1% ZIRCONIUM

1800°F

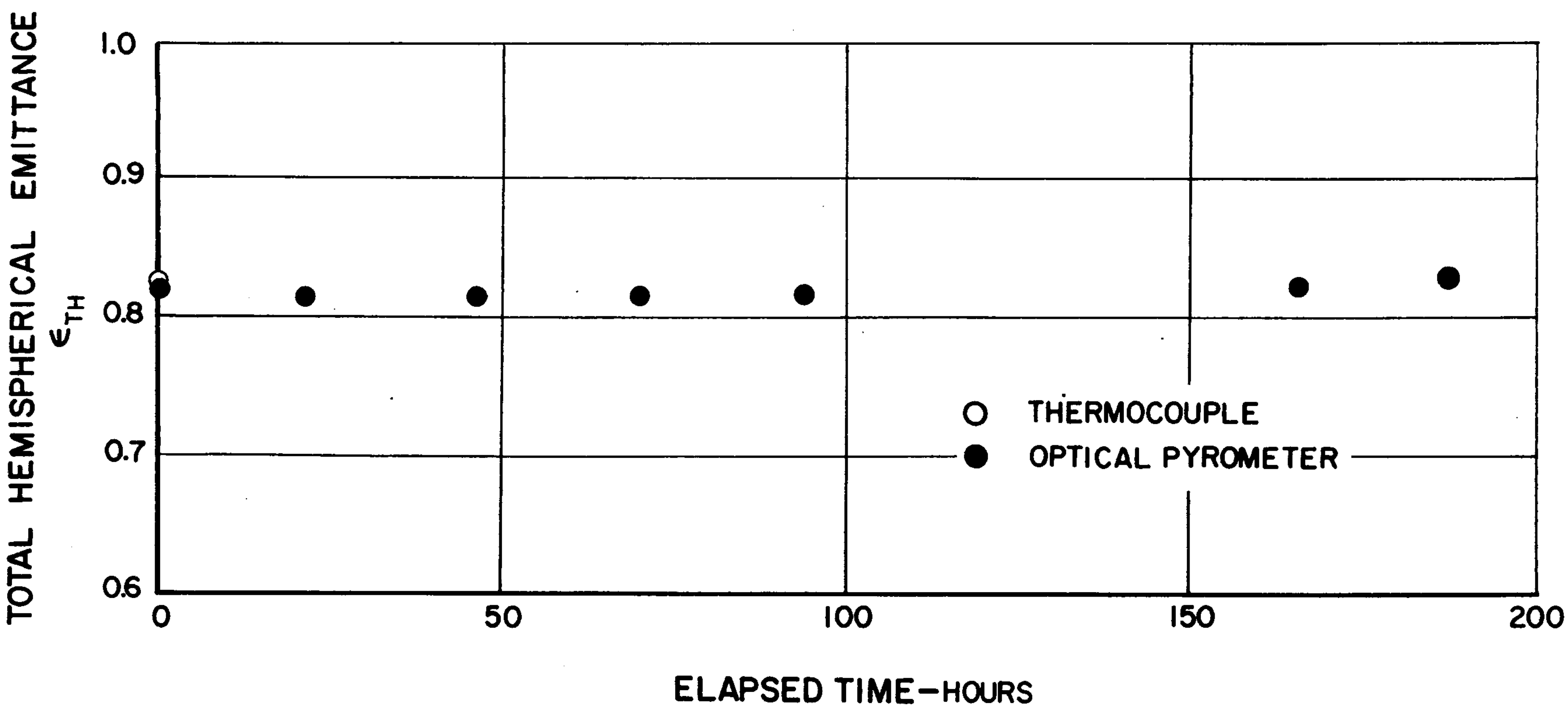


Figure 22

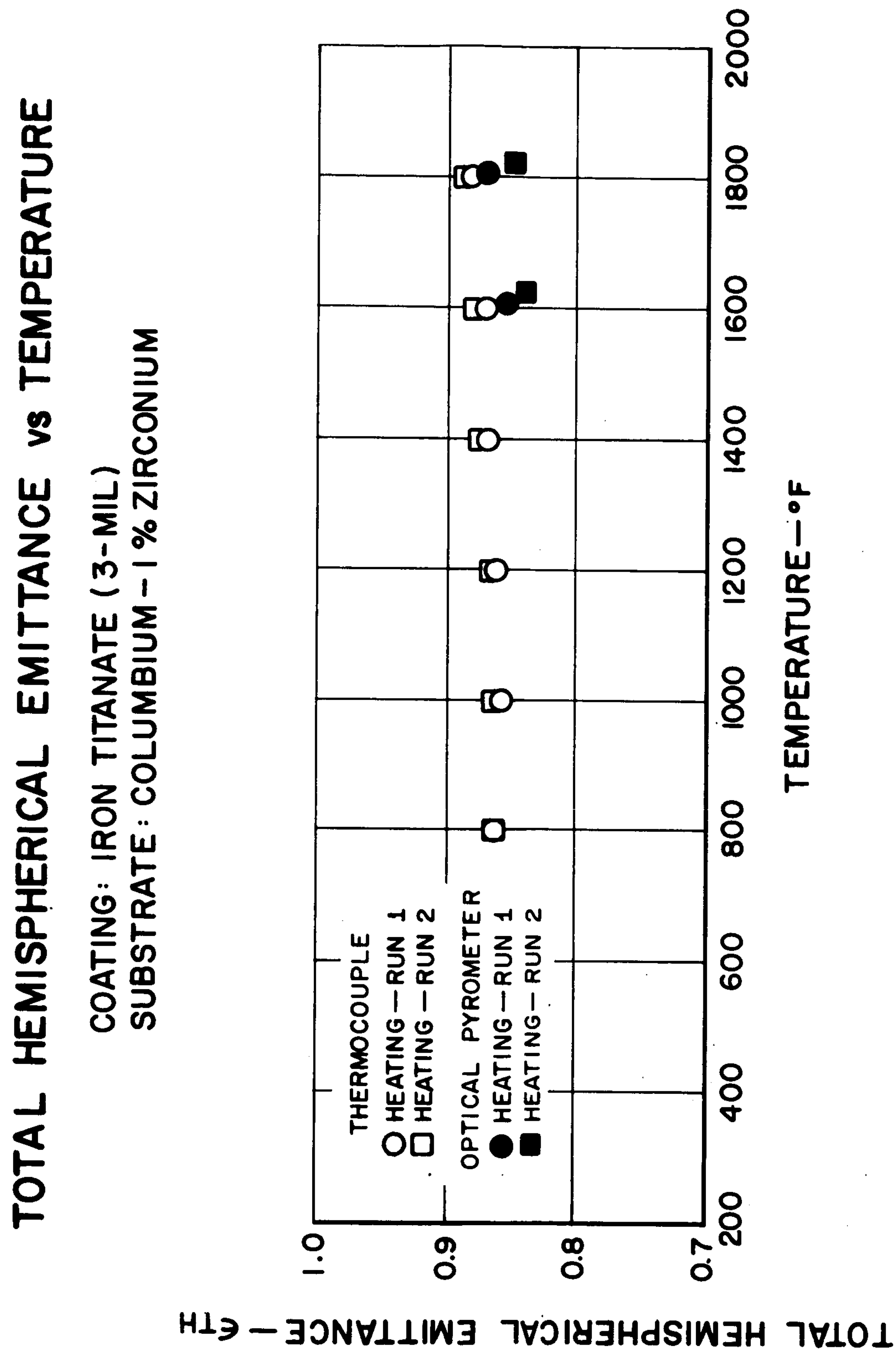

TOTAL HEMISPHERICAL EMITTANCE vs TEMPERATURE
COATING: IRON TITANATE (3-MIL)
SUBSTRATE: COLUMBIUM - 1 % ZIRCONIUM
THERMOCOUPLE
HEATING — RUN 1
HEATING — RUN 2
OPTICAL PYROMETER
HEATING — RUN 1
HEATING — RUN 2
1.0
0.9
0.8
0.7
200
400
600
800
1000
1200
1400
1600
1800
2000
TEMPERATURE — °F
TOTAL HEMISPHERICAL EMITTANCE — ϵTH

Figure 23

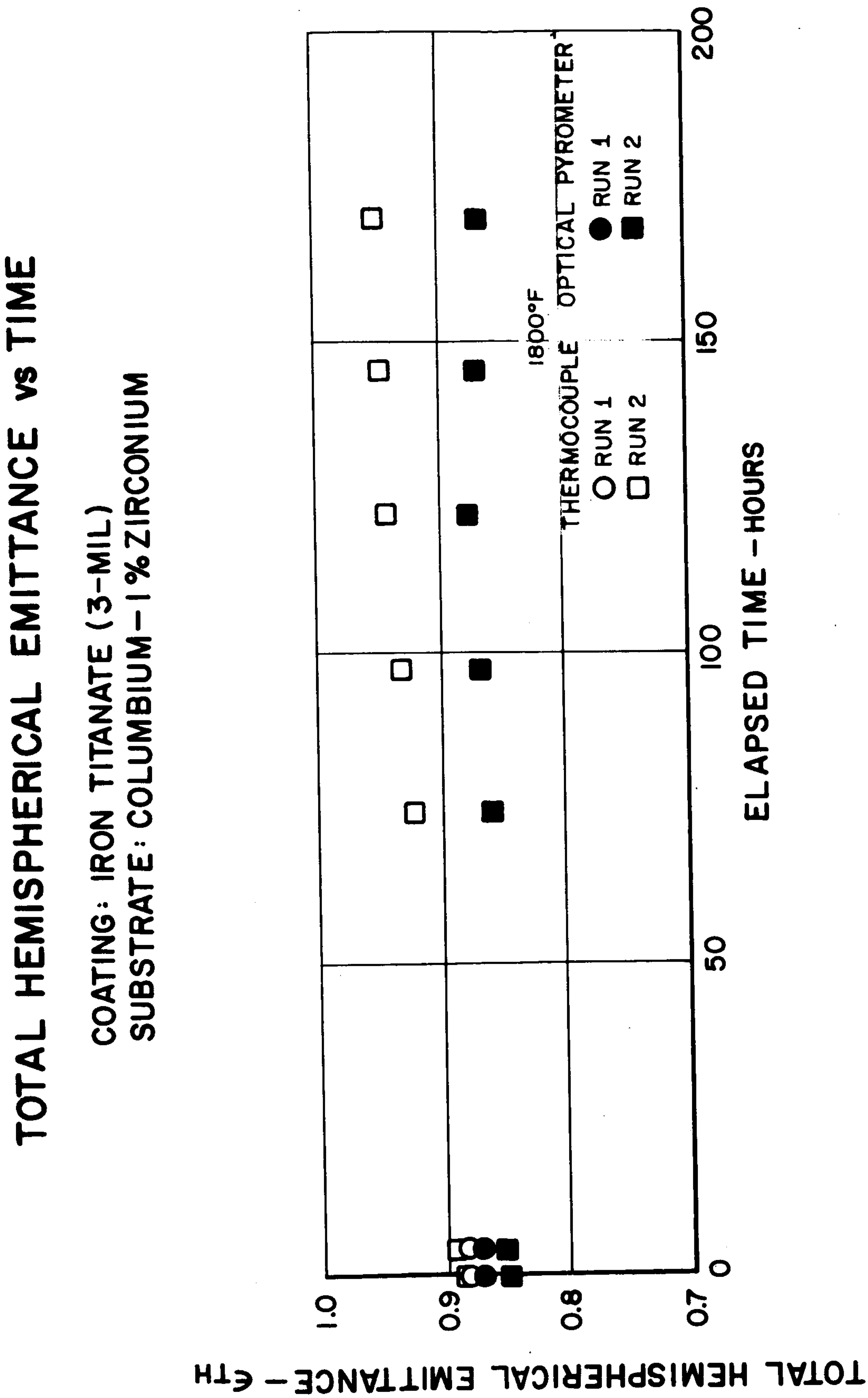

TOTAL HEMISPHERICAL EMITTANCE vs TIME
COATING: IRON TITANATE (3-MIL)
SUBSTRATE: COLUMBIUM-1% ZIRCONIUM
1800°F
THERMOCOUPLE
RUN 1
RUN 2
OPTICAL PYROMETER
RUN 1
RUN 2
0
50
100
150
200
ELAPSED TIME-HOURS
1.0
0.9
0.8
0.7
TOTAL HEMISPHERICAL EMITTANCE - ϵ_{TH}

Figure 24

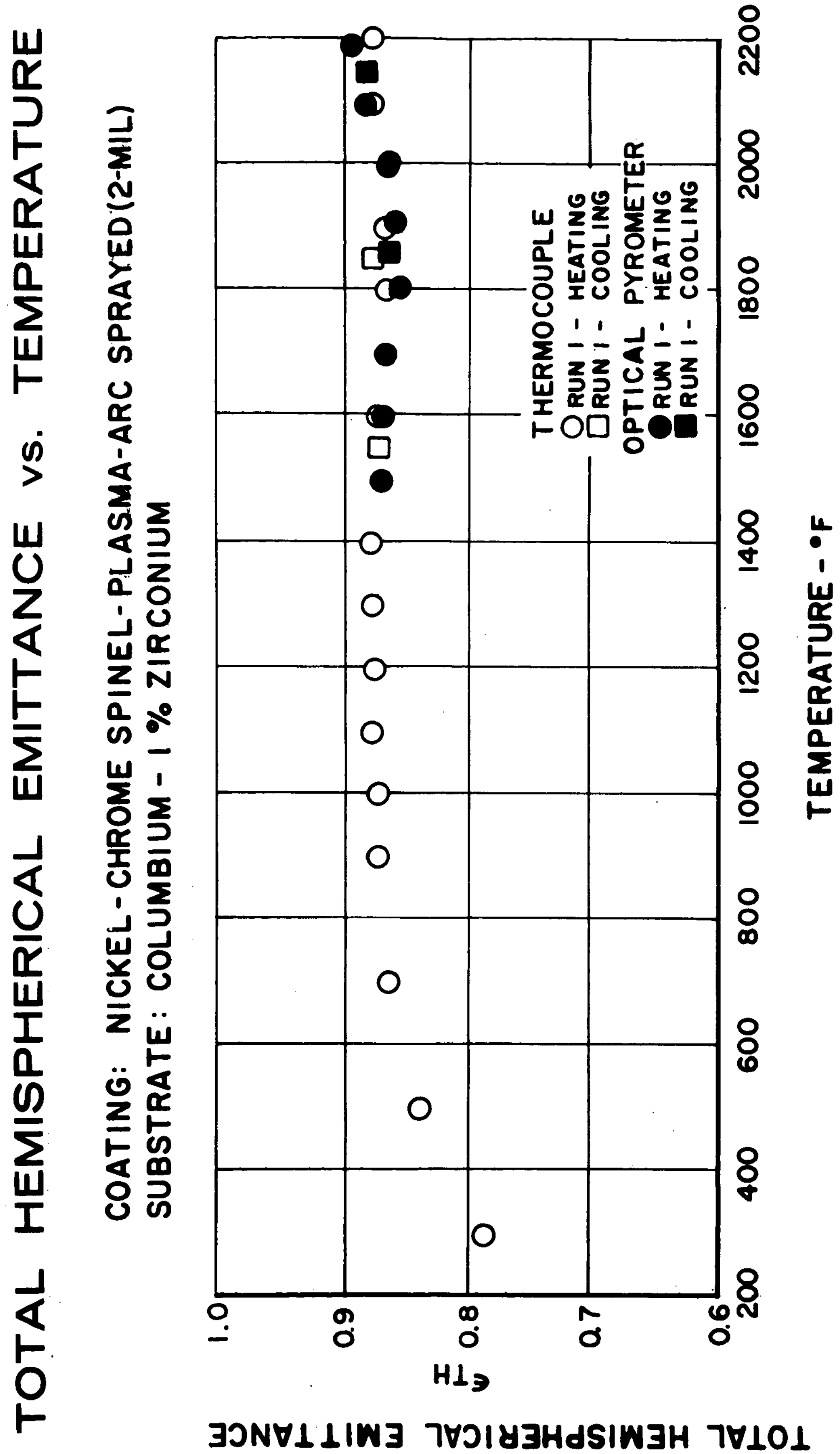

TOTAL HEMISPHERICAL EMITTANCE vs. TEMPERATURE
COATING: NICKEL-CHROME SPINEL-PLASMA-ARC SPRAYED (2-MIL)
SUBSTRATE: COLUMBIUM - 1 % ZIRCONIUM
THERMOCOUPLE
RUN I - HEATING
RUN I - COOLING
OPTICAL PYROMETER
RUN I - HEATING
RUN I - COOLING
1.0
0.9
0.8
0.7
0.6
ϵ_{TH}
TOTAL HEMISPHERICAL EMITTANCE
200
400
600
800
1000
1200
1400
1600
1800
2000
2200
TEMPERATURE - °F

Figure 25

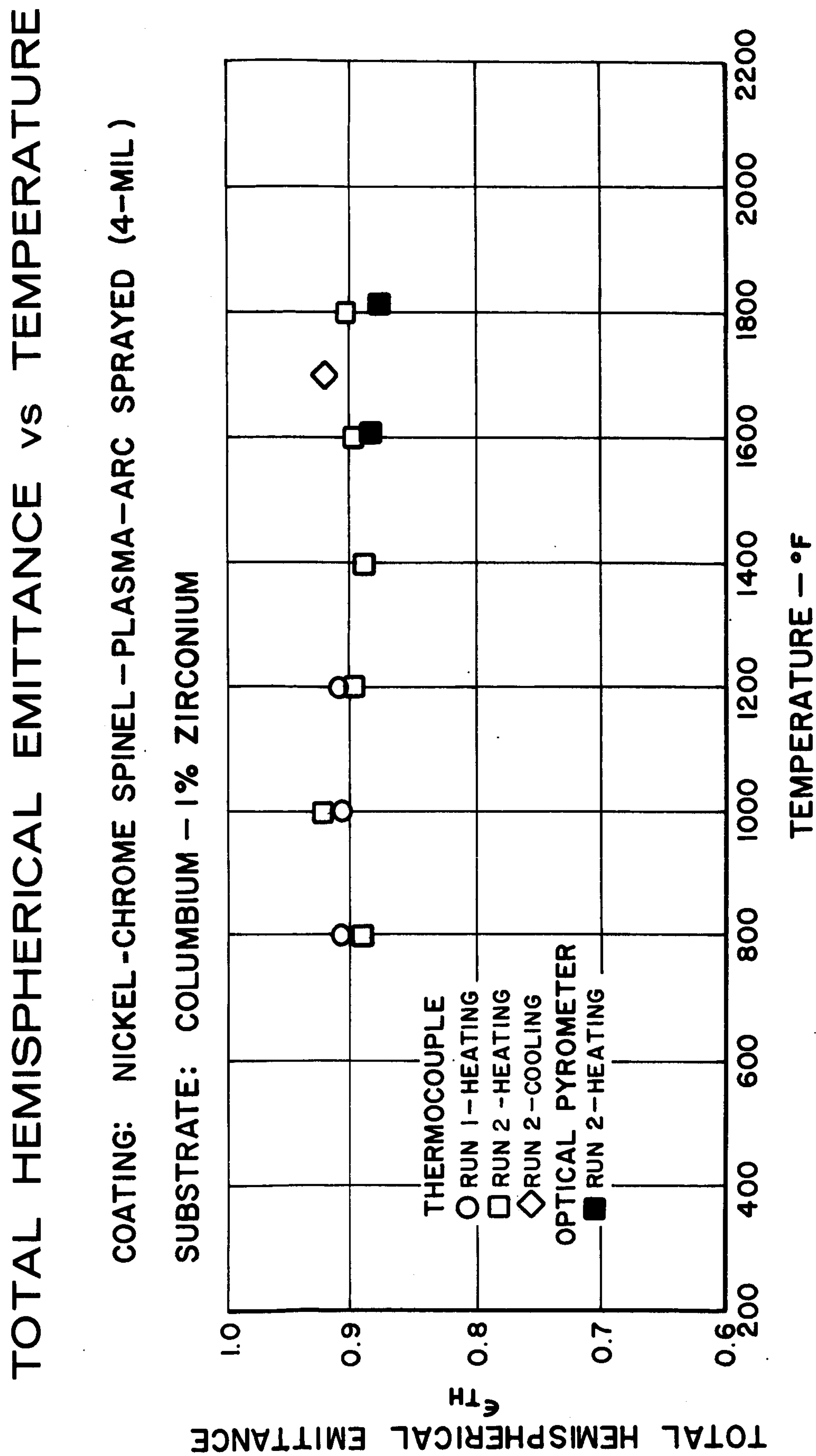

TOTAL HEMISPHERICAL EMITTANCE vs TEMPERATURE
COATING: NICKEL-CHROME SPINEL-PLASMA-ARC SPRAYED (4-MIL)
SUBSTRATE: COLUMBIUM - 1% ZIRCONIUM
THERMOCOUPLE
RUN 1-HEATING
RUN 2-HEATING
RUN 2-COOLING
OPTICAL PYROMETER
RUN 2-HEATING
TOTAL HEMISPHERICAL EMITTANCE
ϵ_{TH}
1.0
0.9
0.8
0.7
0.6
200
400
600
800
1000
1200
1400
1600
1800
2000
2200
TEMPERATURE — °F

Figure 26

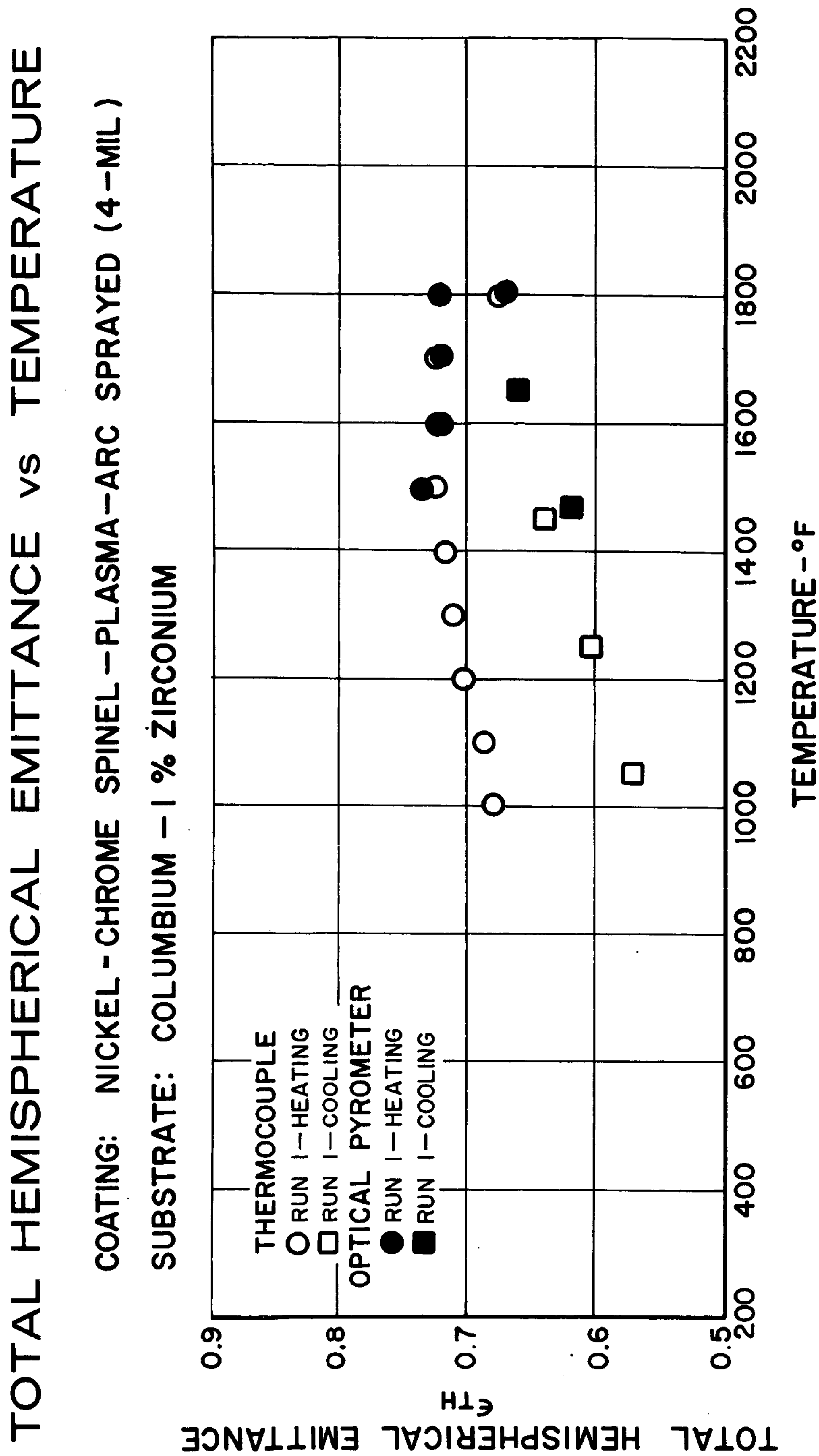

TOTAL HEMISPHERICAL EMITTANCE vs TEMPERATURE
COATING: NICKEL-CHROME SPINEL-PLASMA-ARC SPRAYED (4-MIL)
SUBSTRATE: COLUMBIUM - 1 % ZIRCONIUM
THERMOCOUPLE
RUN 1—HEATING
RUN 1—COOLING
OPTICAL PYROMETER
RUN 1—HEATING
RUN 1—COOLING
TOTAL HEMISPHERICAL EMITTANCE
ϵ_{TH}
0.9
0.8
0.7
0.6
0.5
200
400
600
800
1000
1200
1400
1600
1800
2000
2200
TEMPERATURE-°F

Figure 27

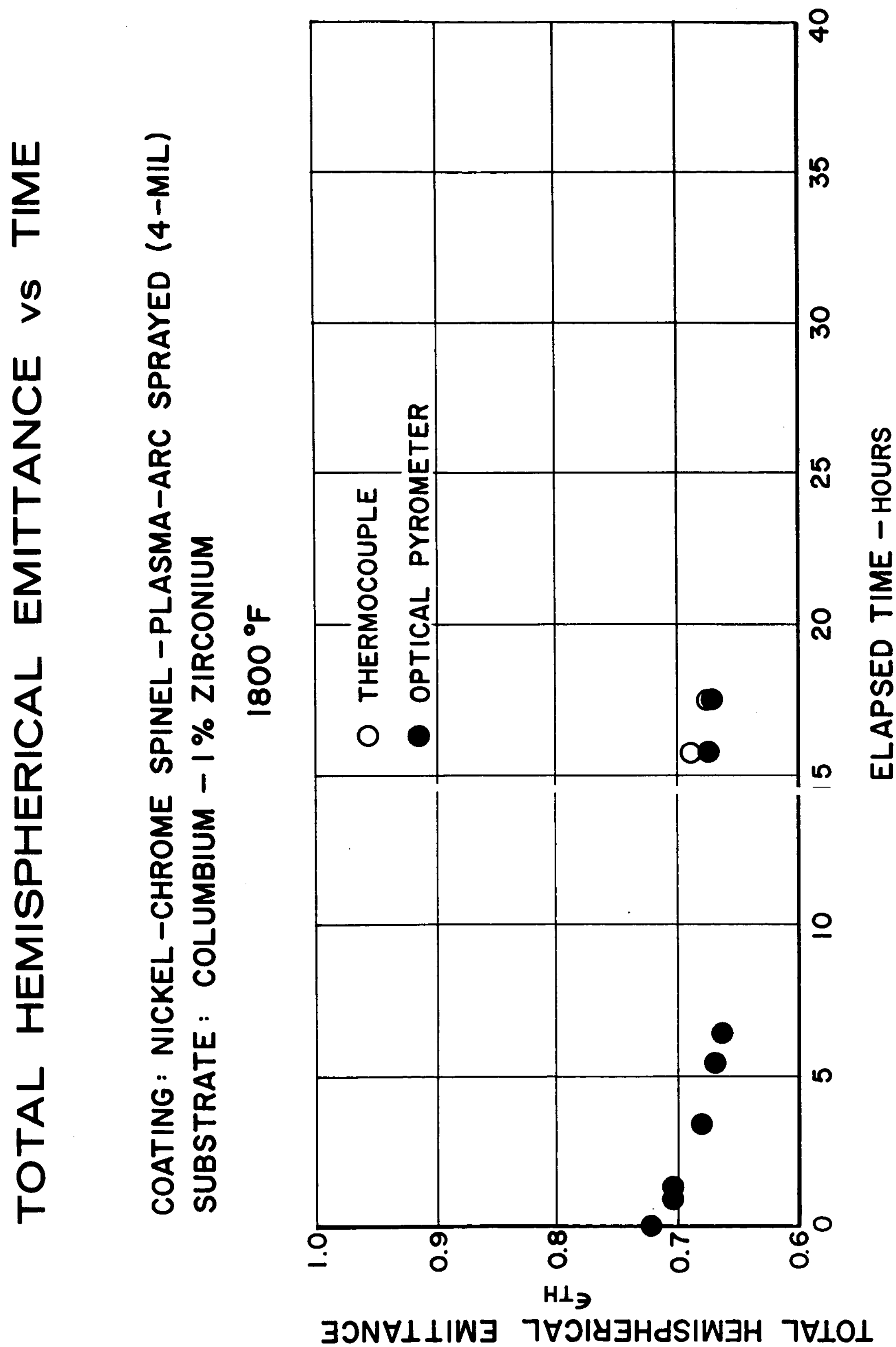

TOTAL HEMISPHERICAL EMITTANCE vs TIME
COATING: NICKEL-CHROME SPINEL-PLASMA-ARC SPRAYED (4-MIL)
SUBSTRATE: COLUMBIUM - 1% ZIRCONIUM
1800°F
○ THERMOCOUPLE
● OPTICAL PYROMETER
TOTAL HEMISPHERICAL EMITTANCE
ϵ_{TH}
1.0
0.9
0.8
0.7
0.6
0
5
10
15
20
25
30
35
40
ELAPSED TIME - HOURS

Figure 28

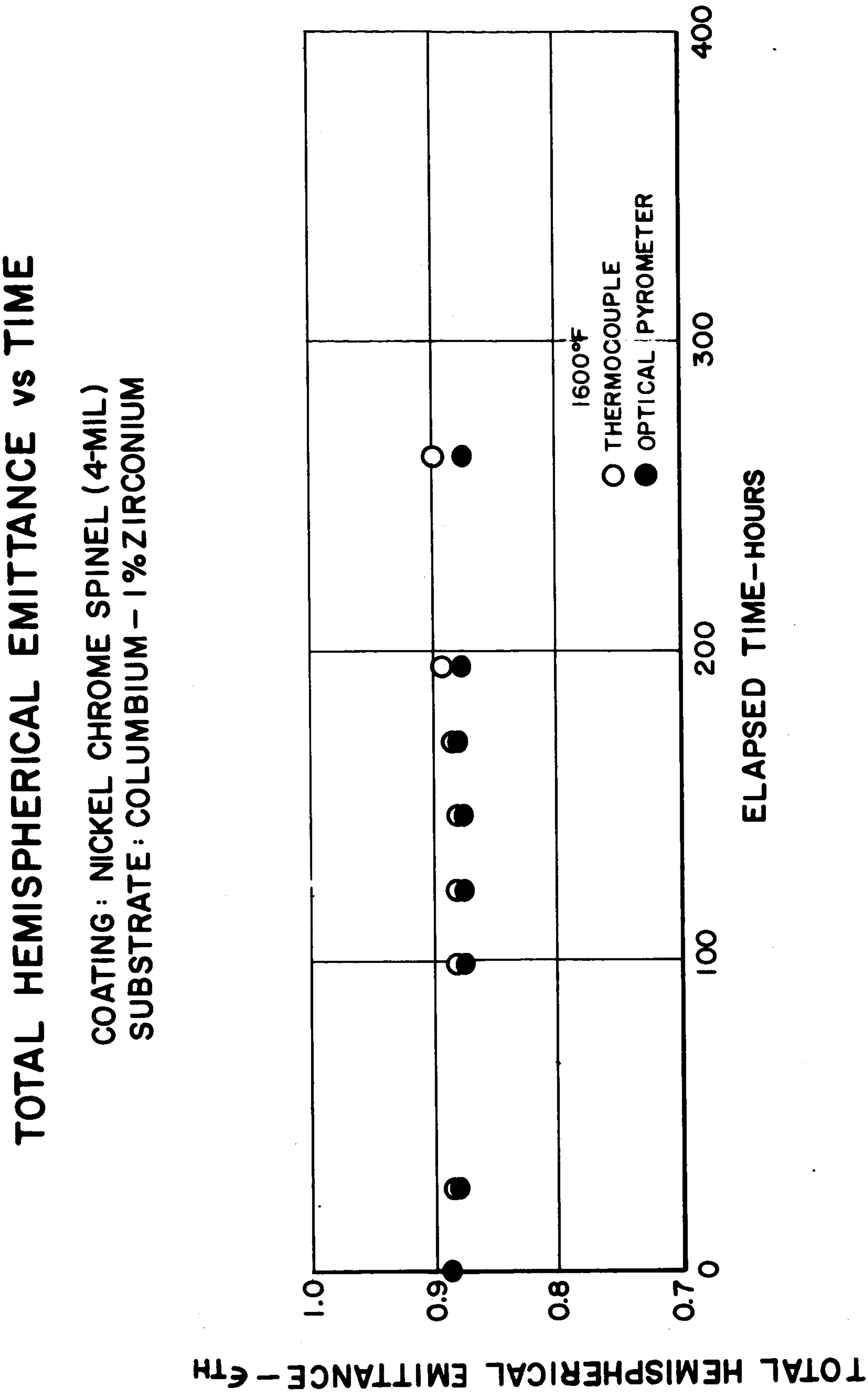

TOTAL HEMISPHERICAL EMITTANCE vs TIME
COATING: NICKEL CHROME SPINEL (4-MIL)
SUBSTRATE: COLUMBIUM – 1% ZIRCONIUM
1600°F
THERMOCOUPLE
OPTICAL PYROMETER
ELAPSED TIME–HOURS
0
100
200
300
400
1.0
0.9
0.8
0.7
TOTAL HEMISPHERICAL EMITTANCE – ε TH

Figure 29

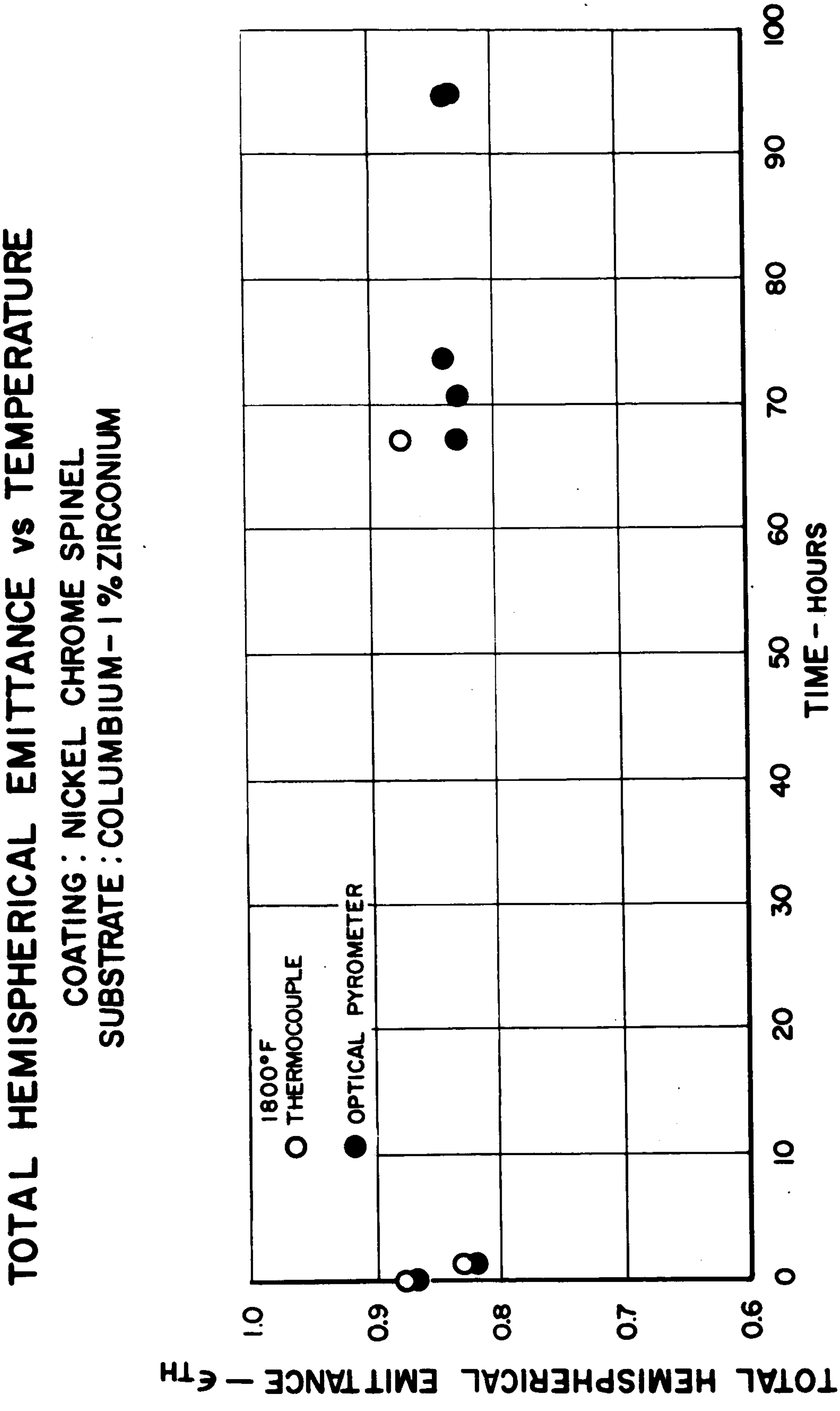

TOTAL HEMISPHERICAL EMITTANCE vs TEMPERATURE
COATING : NICKEL CHROME SPINEL
SUBSTRATE : COLUMBIUM - 1 % ZIRCONIUM
1800°F
THERMOCOUPLE
OPTICAL PYROMETER
0
10
20
30
40
50
60
70
80
90
100
TIME - HOURS
1.0
0.9
0.8
0.7
0.6
TOTAL HEMISPHERICAL EMITTANCE — ϵ_{TH}

Figure 30

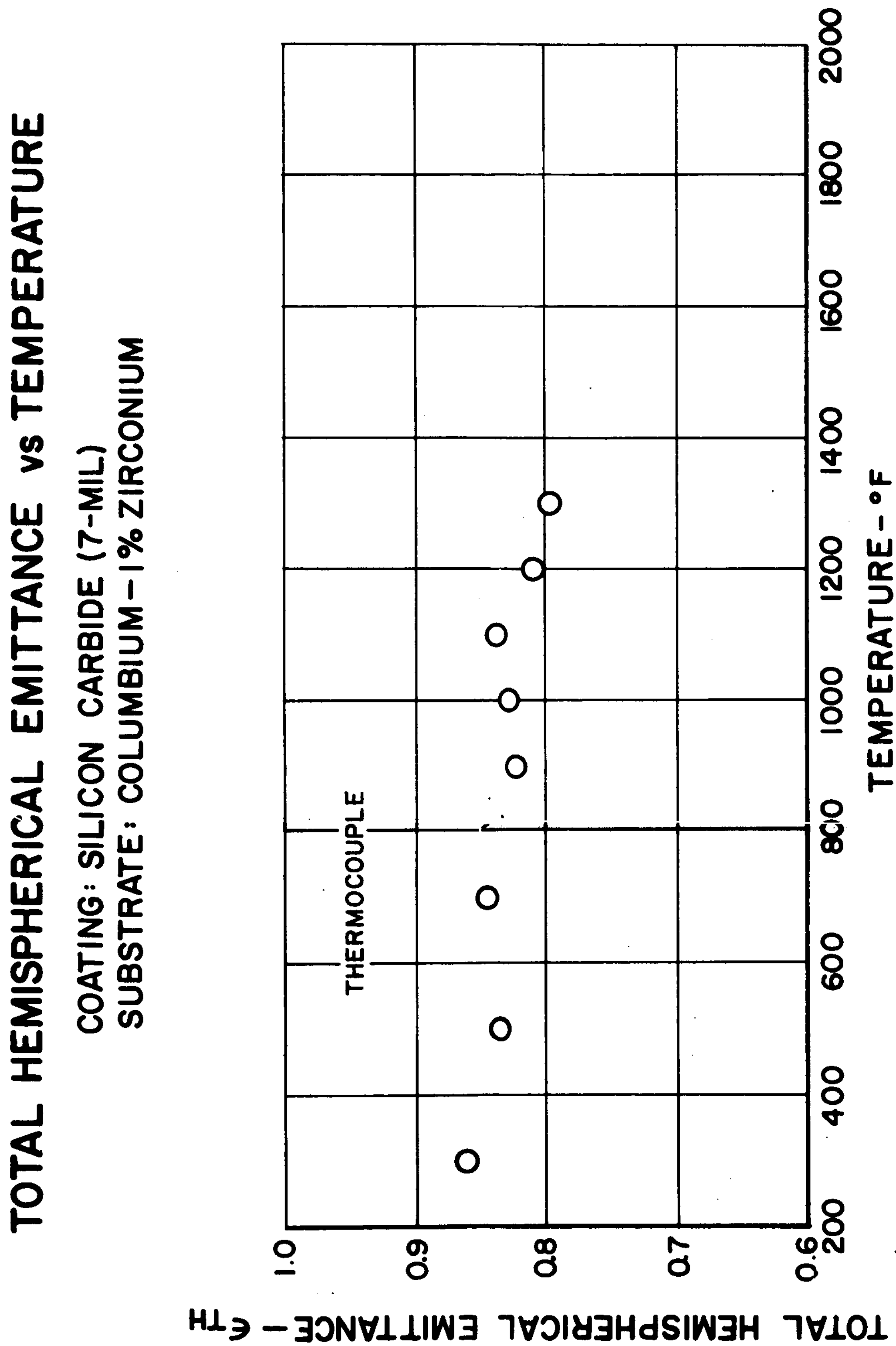

TOTAL HEMISPHERICAL EMITTANCE vs TEMPERATURE
COATING: SILICON CARBIDE (7-MIL)
SUBSTRATE: COLUMBIUM-1% ZIRCONIUM
THERMOCOUPLE
1.0
0.9
0.8
0.7
0.6
200
400
600
800
1000
1200
1400
1600
1800
2000
TEMPERATURE - °F
TOTAL HEMISPHERICAL EMITTANCE — ϵ_{TH}

Figure 31

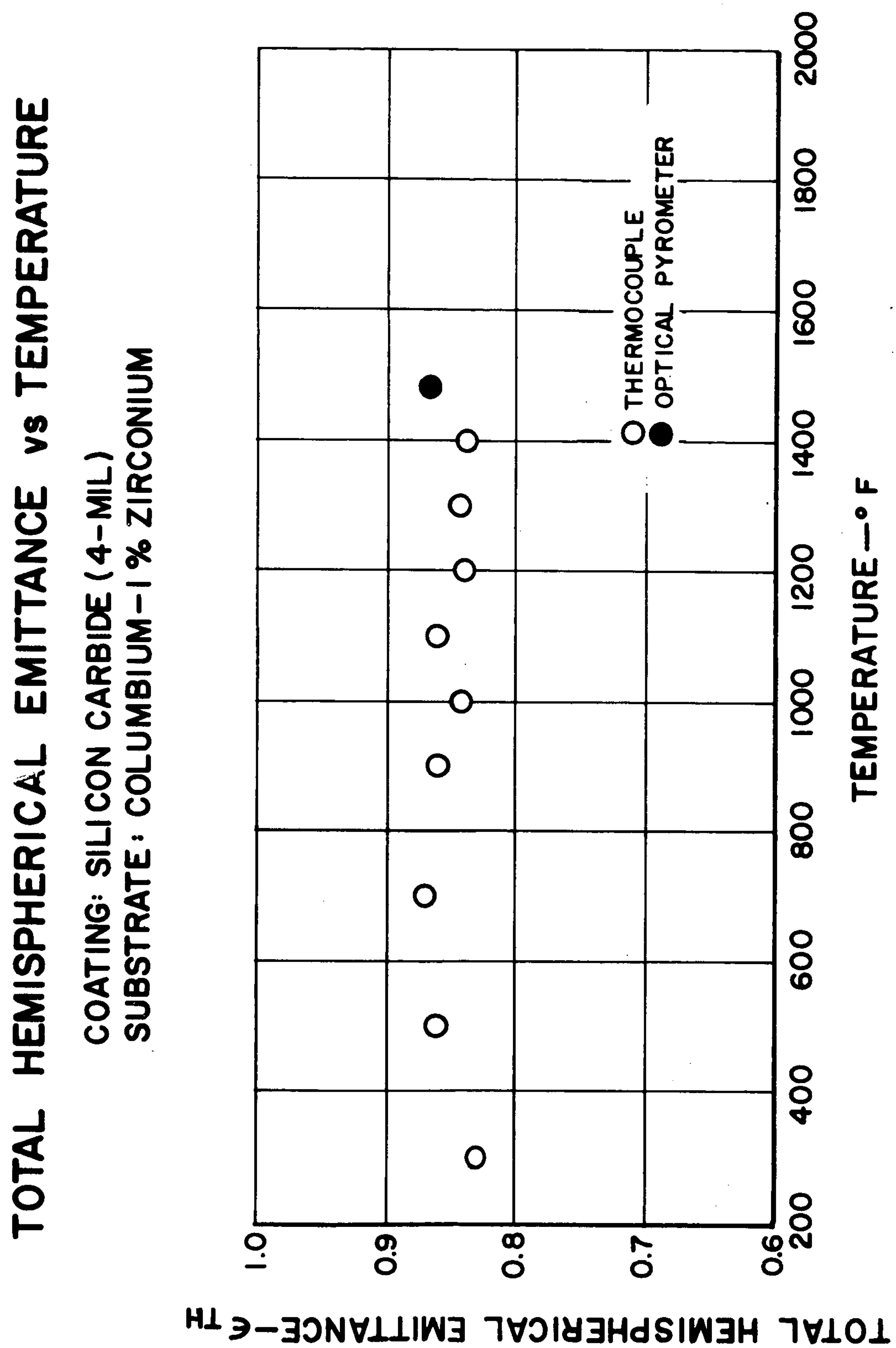

TOTAL HEMISPHERICAL EMITTANCE vs TEMPERATURE
COATING: SILICON CARBIDE (4-MIL)
SUBSTRATE: COLUMBIUM-1% ZIRCONIUM
THERMOCOUPLE
OPTICAL PYROMETER
TOTAL HEMISPHERICAL EMITTANCE—ϵ_{TH}
1.0
0.9
0.8
0.7
0.6
TEMPERATURE—°F
200
400
600
800
1000
1200
1400
1600
1800
2000

Figure 32

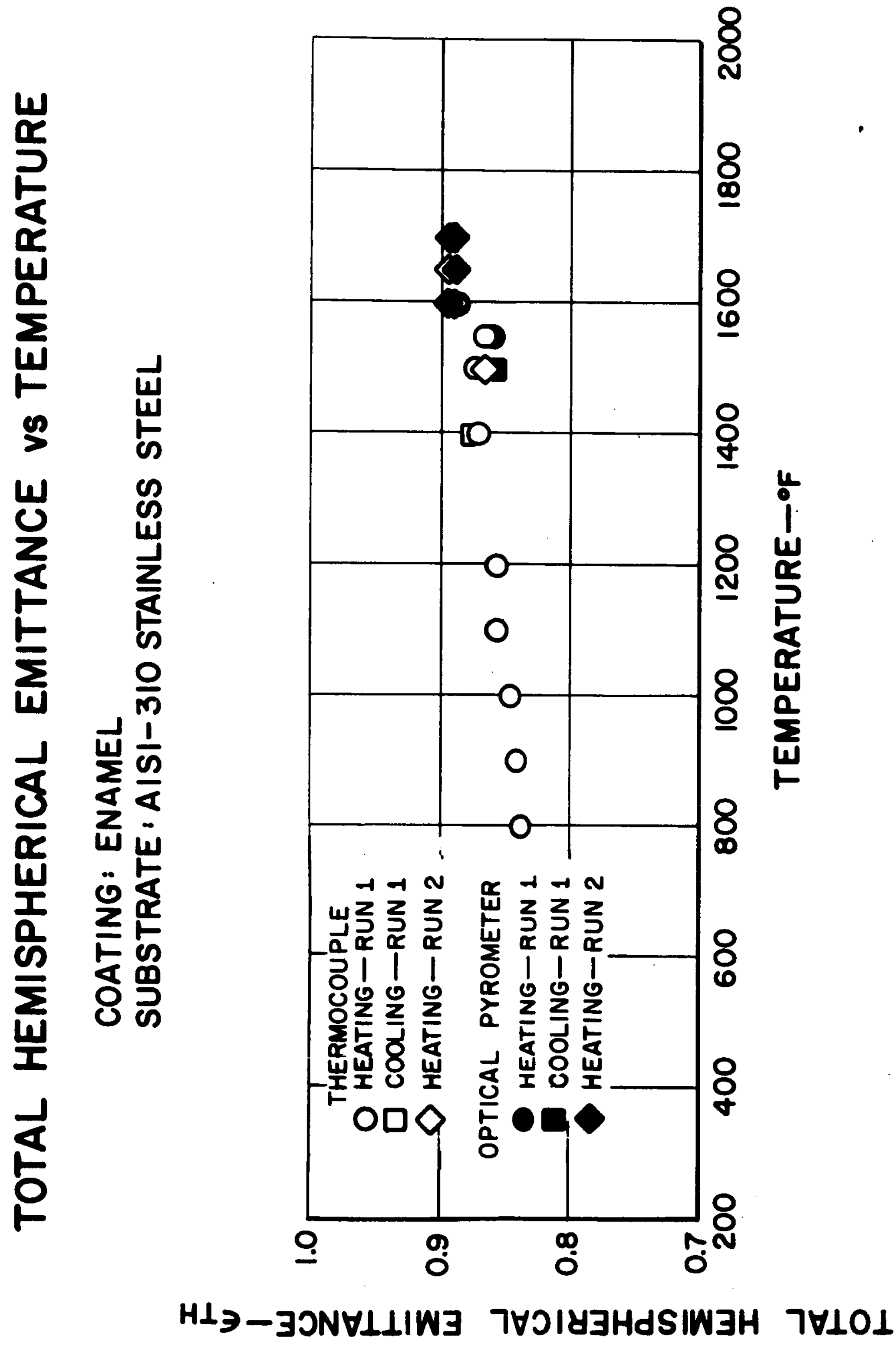

TOTAL HEMISPHERICAL EMITTANCE vs TEMPERATURE
COATING: ENAMEL
SUBSTRATE: AISI-310 STAINLESS STEEL
THERMOCOUPLE
HEATING—RUN 1
COOLING—RUN 1
HEATING—RUN 2
OPTICAL PYROMETER
HEATING—RUN 1
COOLING—RUN 1
HEATING—RUN 2
1.0
0.9
0.8
0.7
200
400
600
800
1000
1200
1400
1600
1800
2000
TEMPERATURE—°F
TOTAL HEMISPHERICAL EMITTANCE—ϵ_{TH}

Figure 33

AUXILIARY VACUUM SYSTEM FOR RESIDUAL GAS ANALYSIS

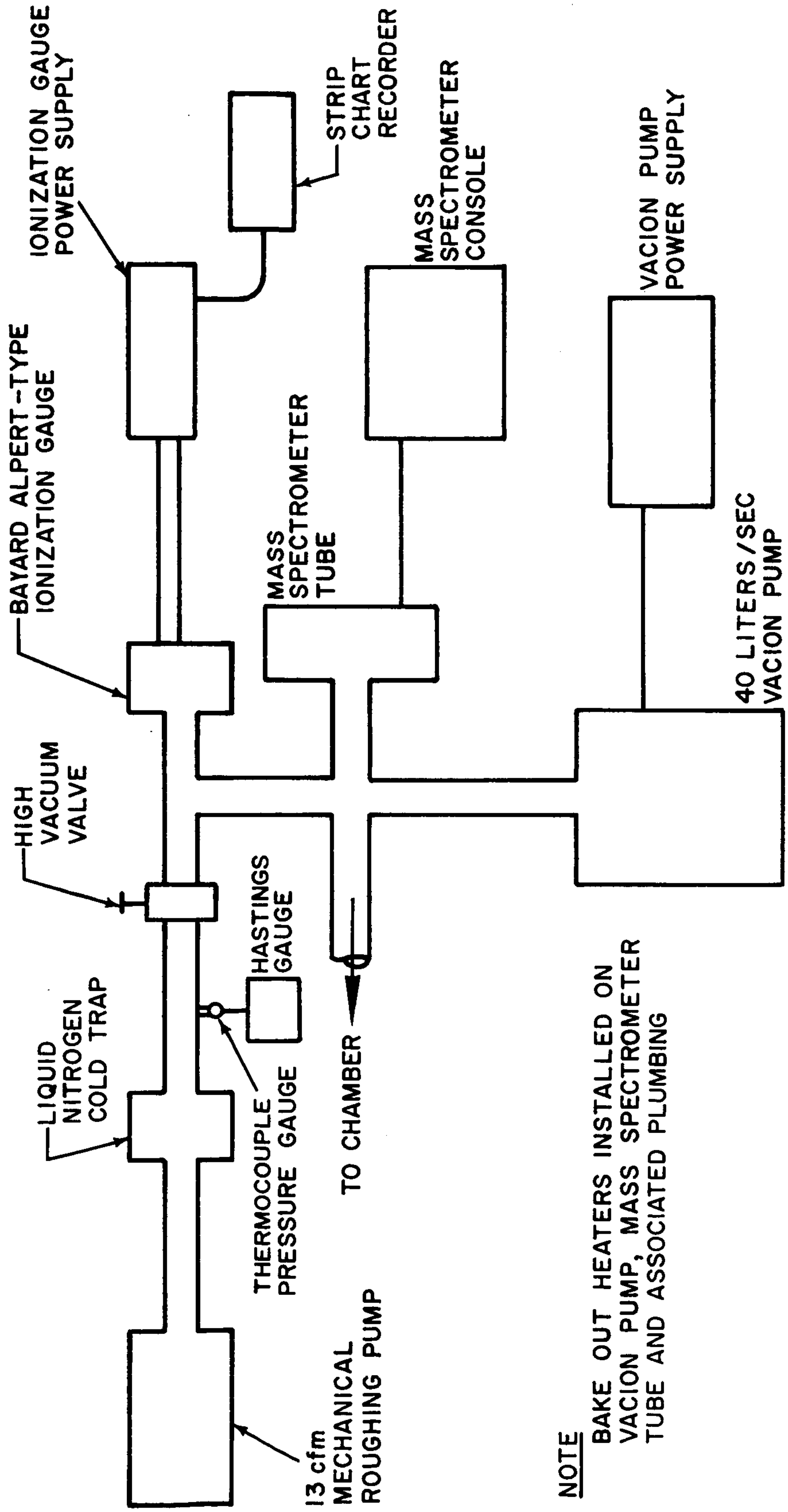
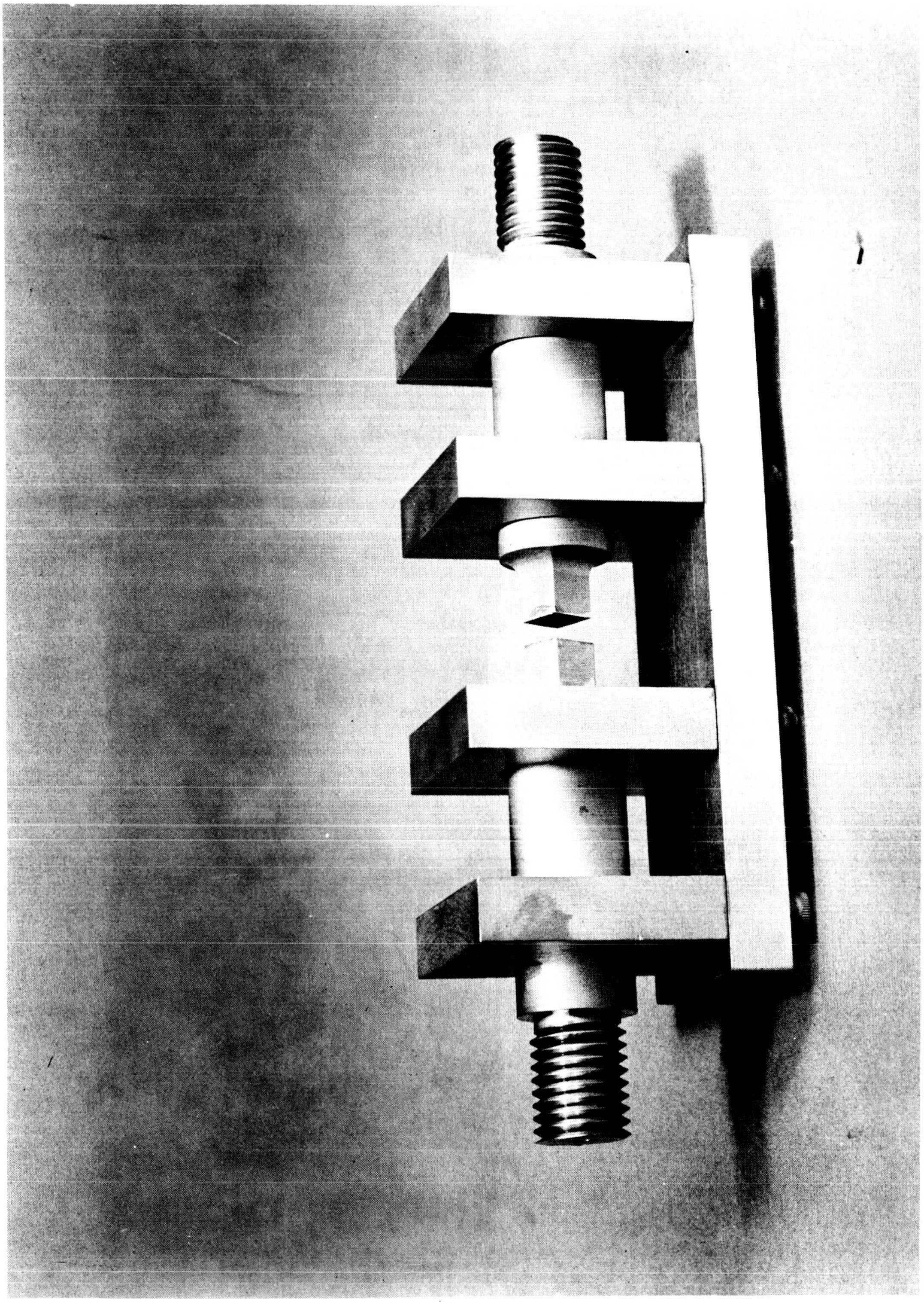
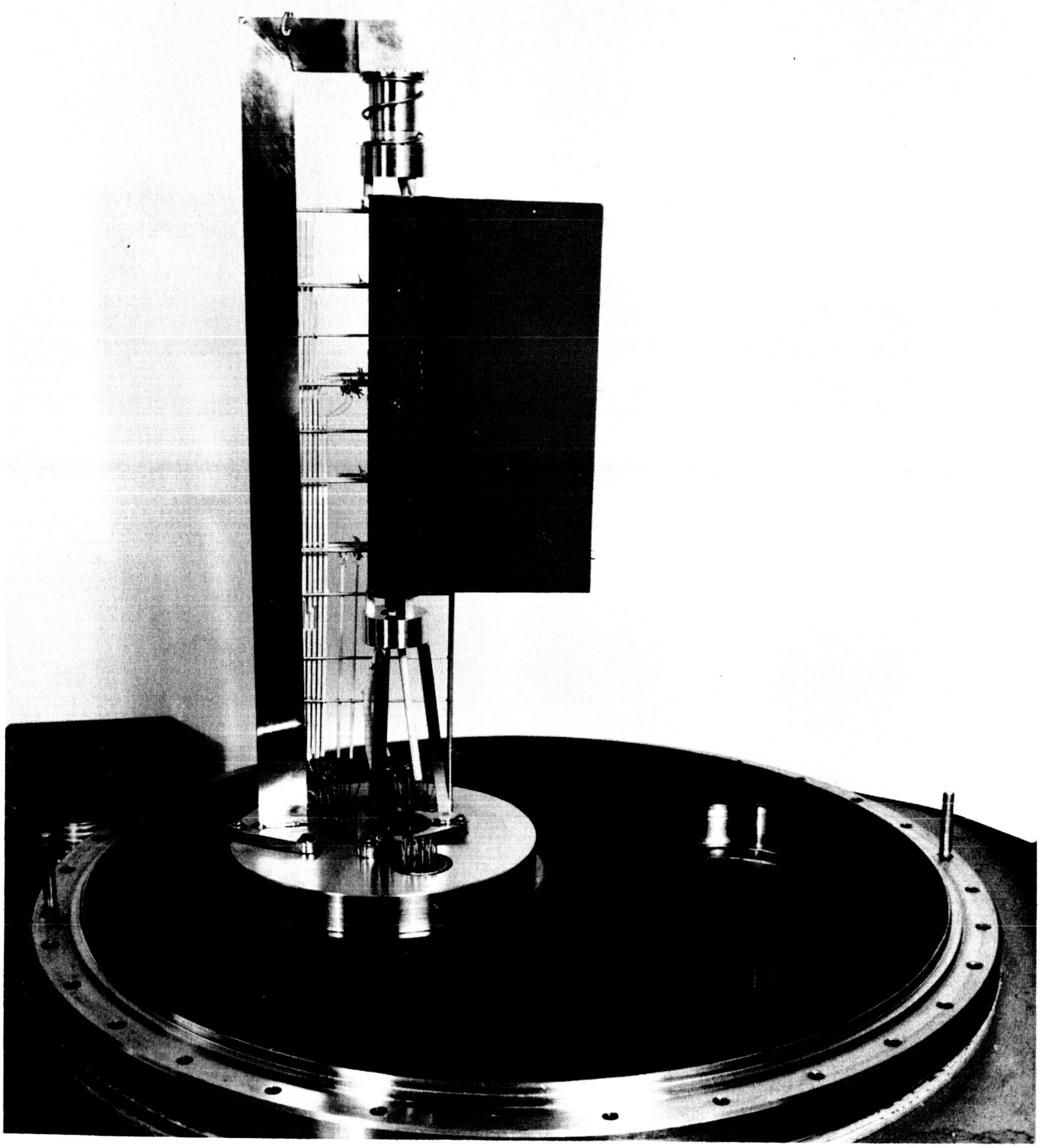


Figure 34



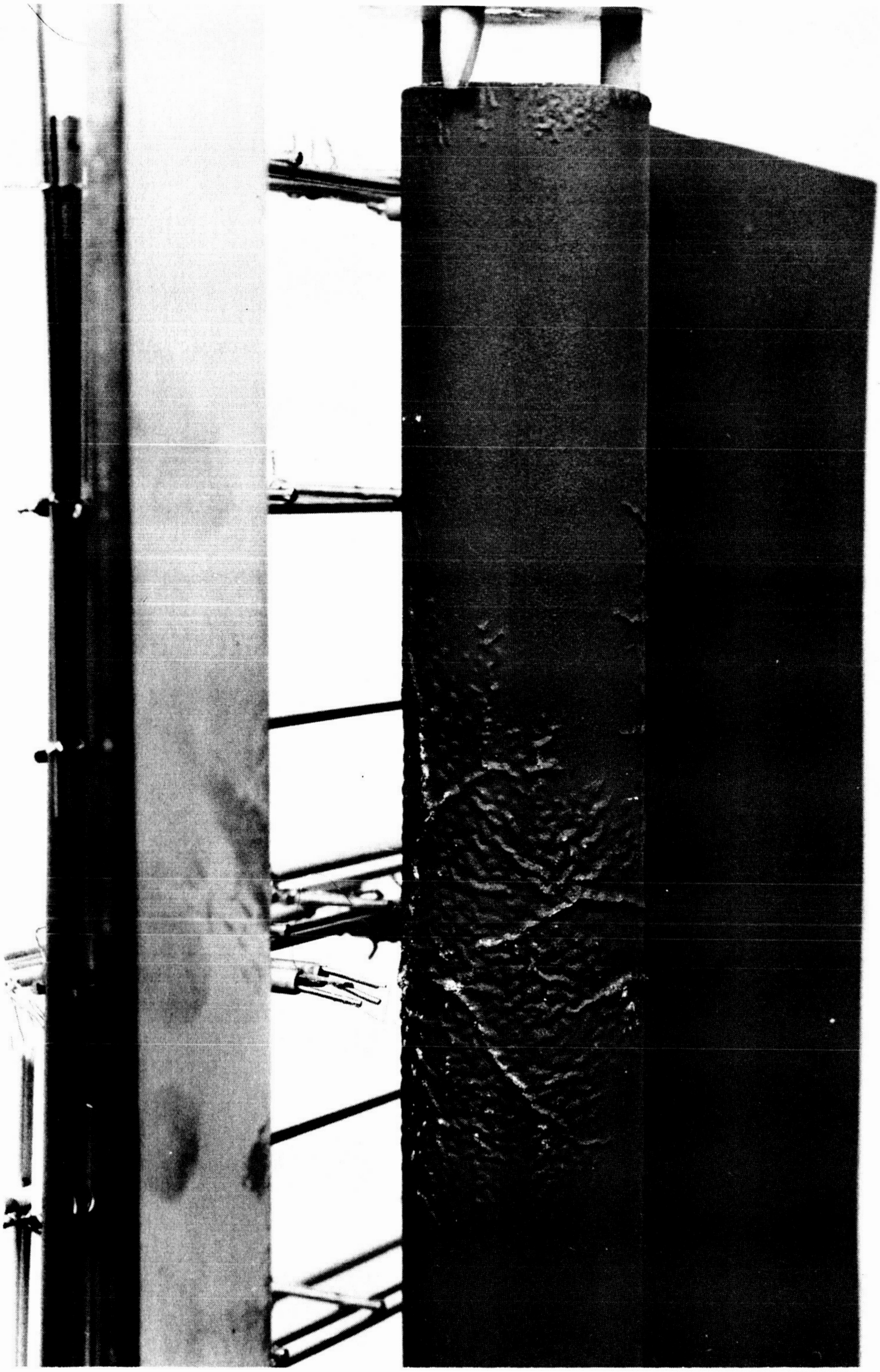
End Grips in Support Used for Bond Testing

Figure 35



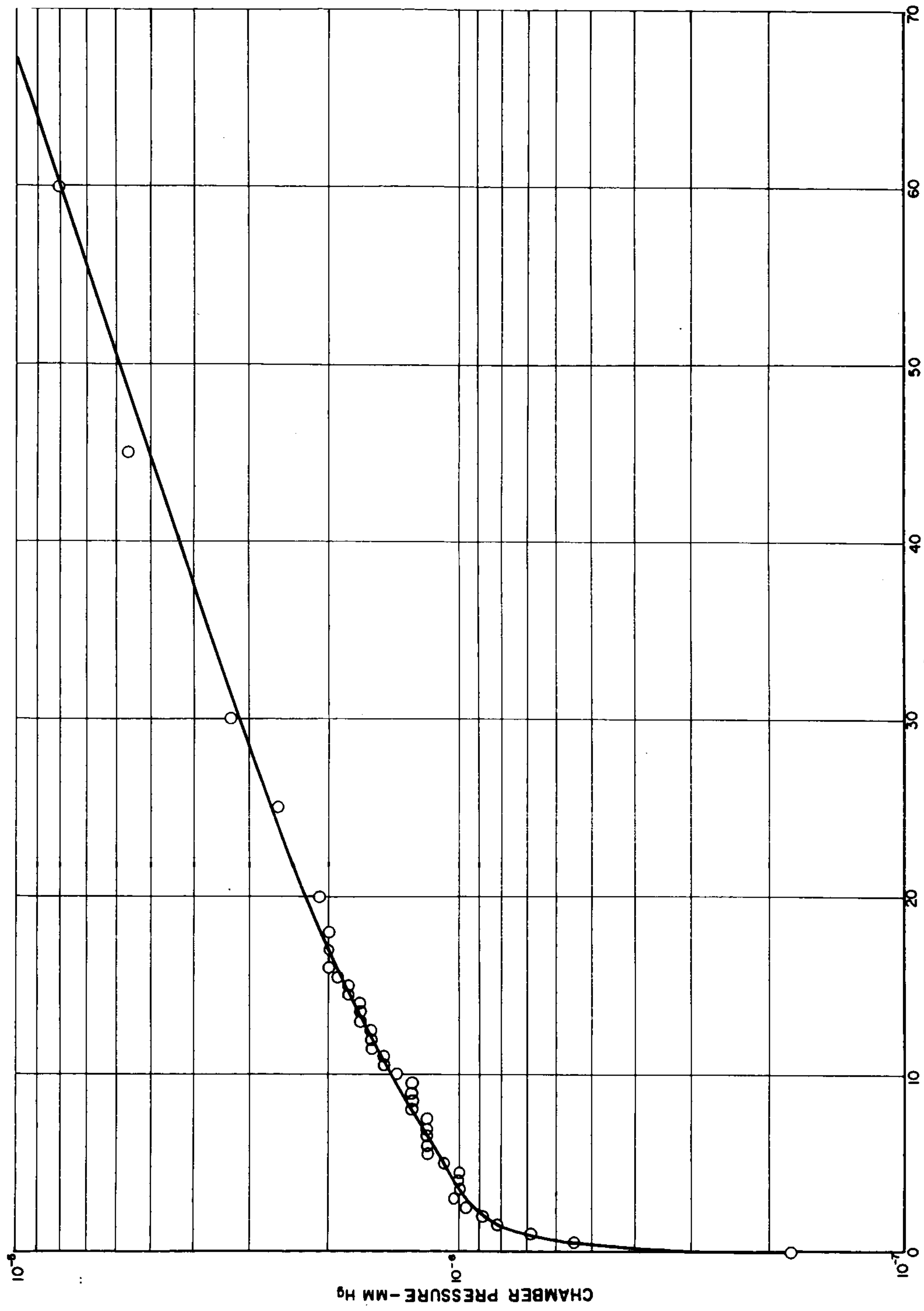
Nickel-Chrome Spinel Coated SNAP-8 Test
Section After Endurance Testing

Figure 36



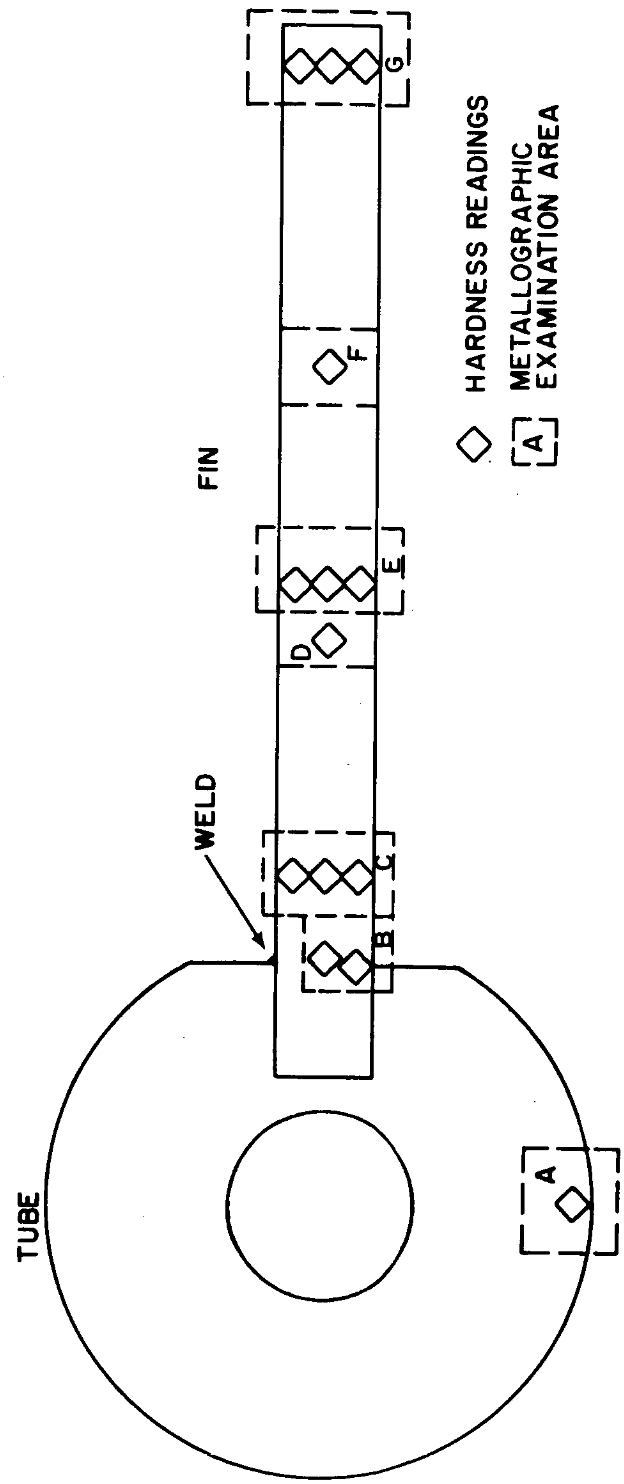
Close-Up View of Wrinkled Portion of Tube on Nickel-Chrome Spinel Coated SNAP-8 Test Section After Endurance Testing

Figure 37



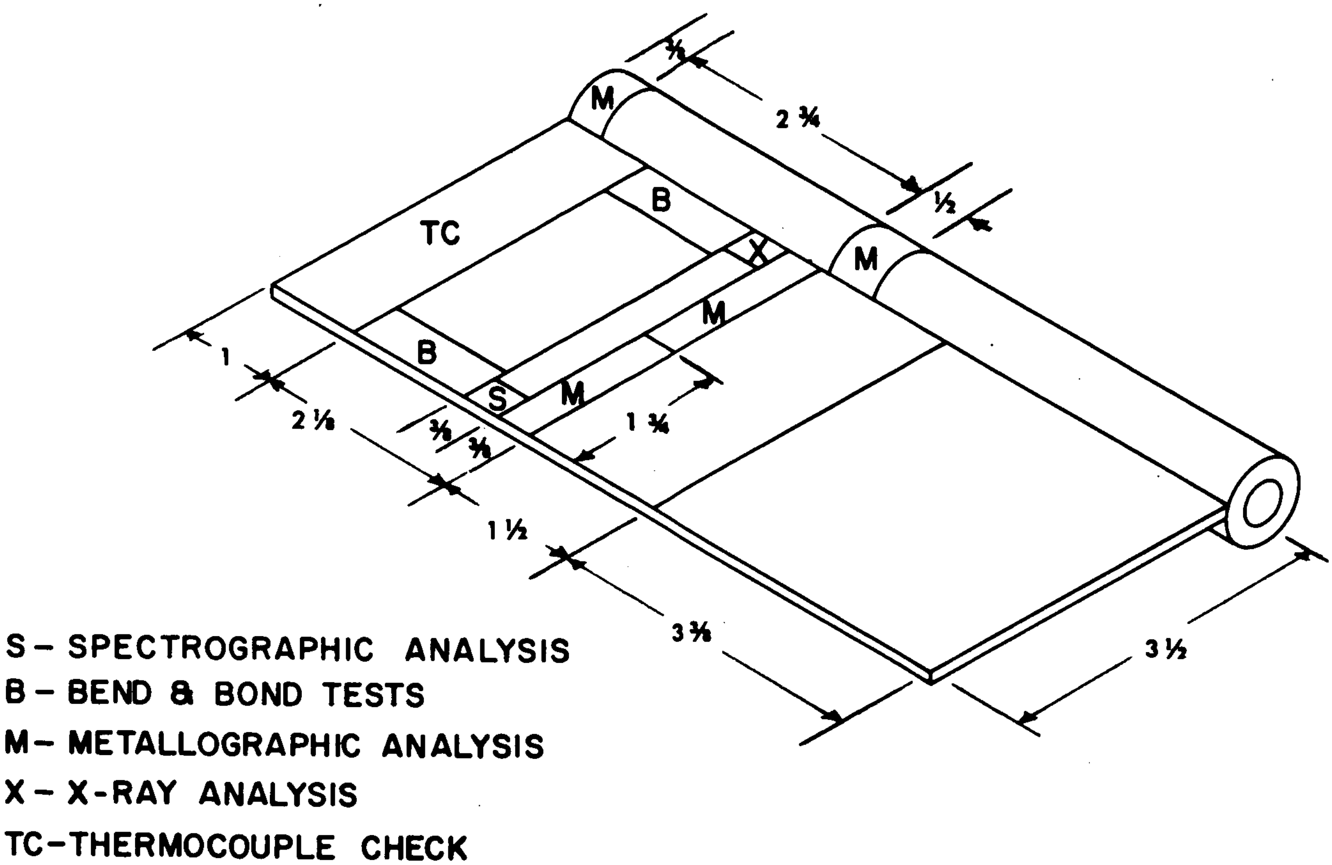
Leak Rate of Test Chamber 1

Figure 38



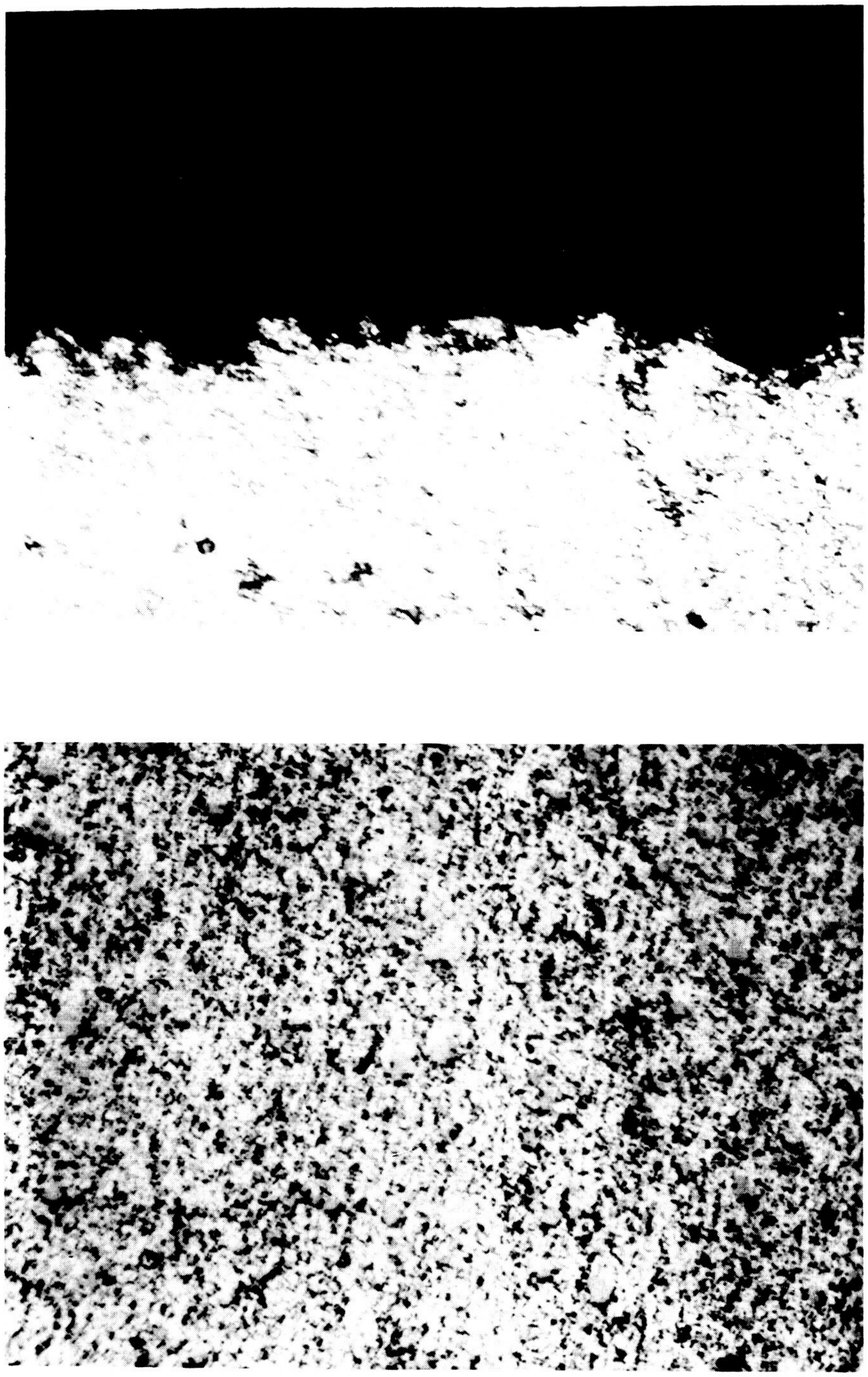
Cross-Section of SNAP-8 Test Section Showing Locations of Hardness Testing and Areas of Metallurgical Examination

Figure 39



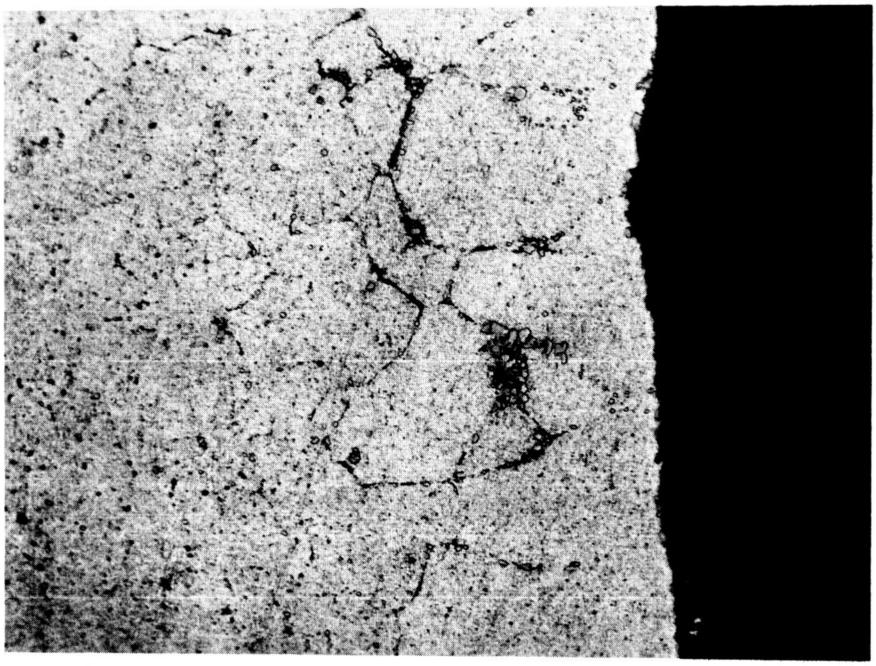
Locations for Samples for Post-Test Analyses of Nickel-Chrome Spinel Coated SNAP-8 Test Section

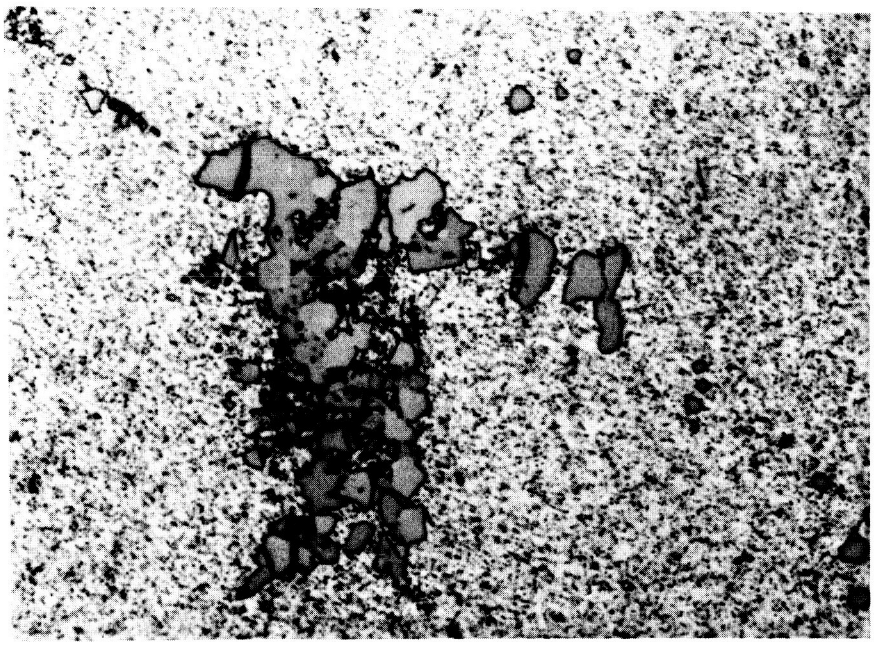
Figure 40



Etchant: 0.5% HF Mag: 500X
Typical Photomicrographs of Fin Portion of Nickel-Chrome Spinel Coated SNAP-8 Test Section Taken at Locations E (Top) and D (Bottom) Shown in Figure 39

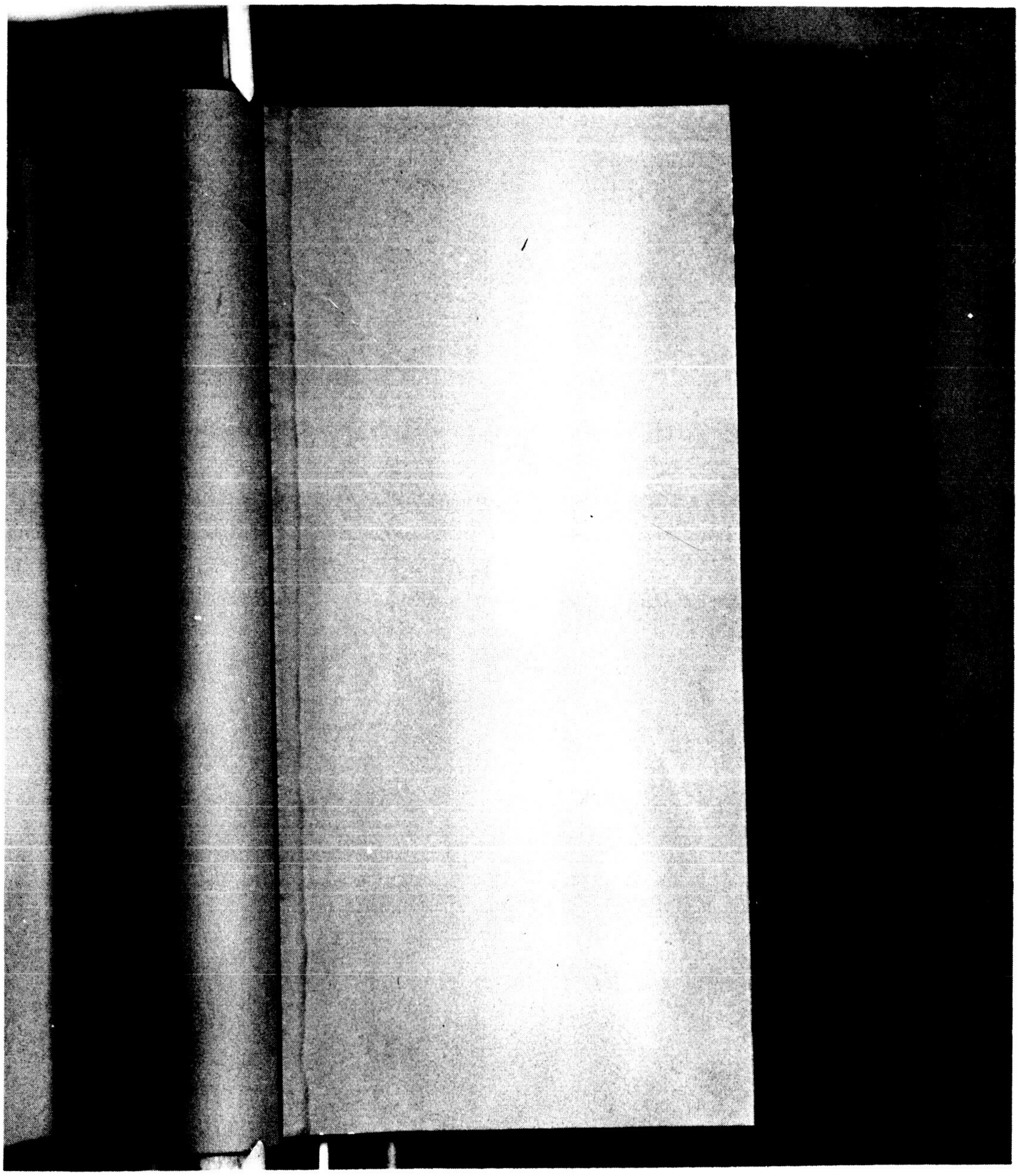
Figure 41





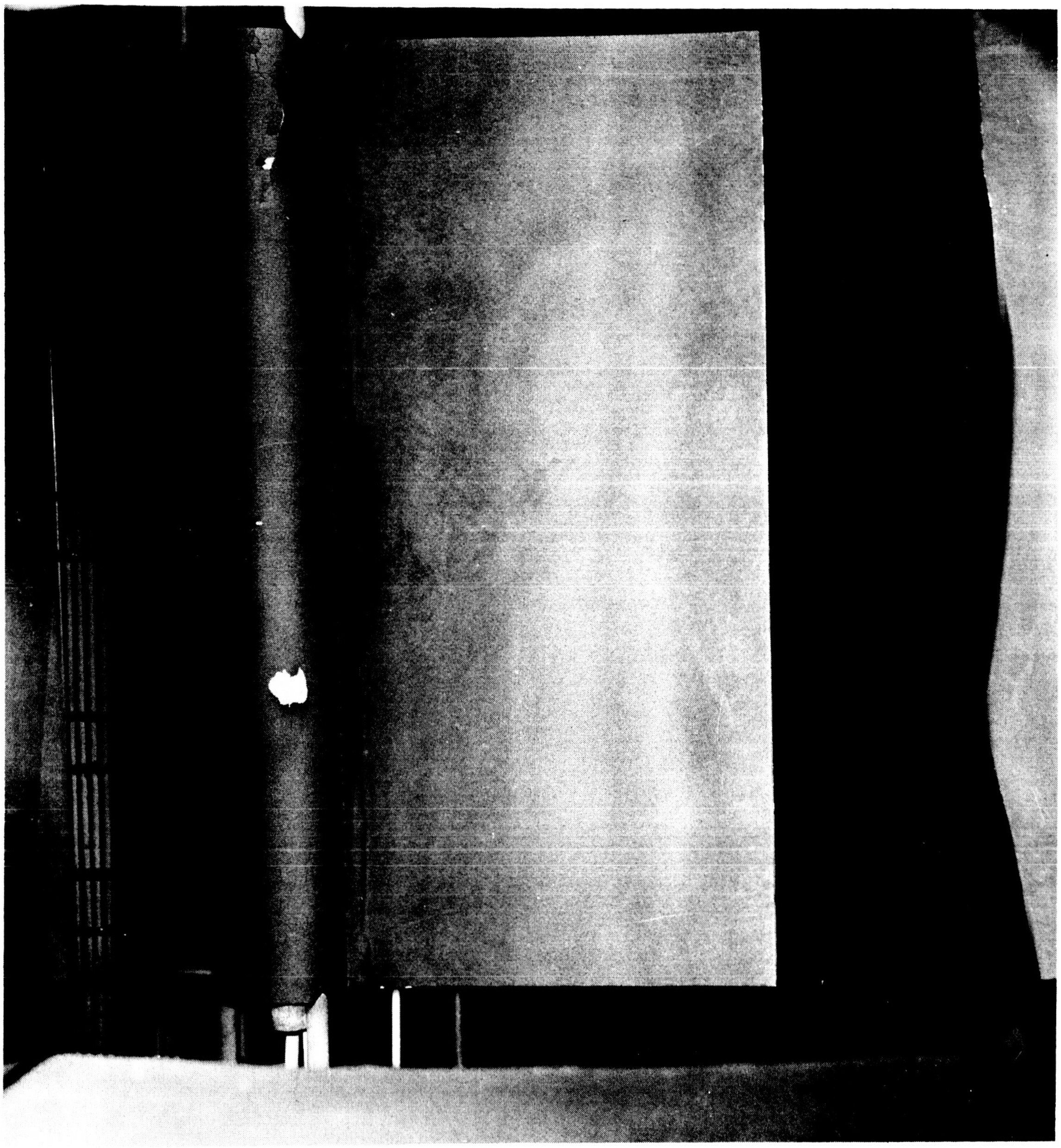
Etchant: 0.5% HF Mag: 100X - Top, 500X - Bottom
Photomicrographs of Wrinkled Portion of Tube on Nickel-Chrome Spinel Coated SNAP-8 Test Section

Figure 42



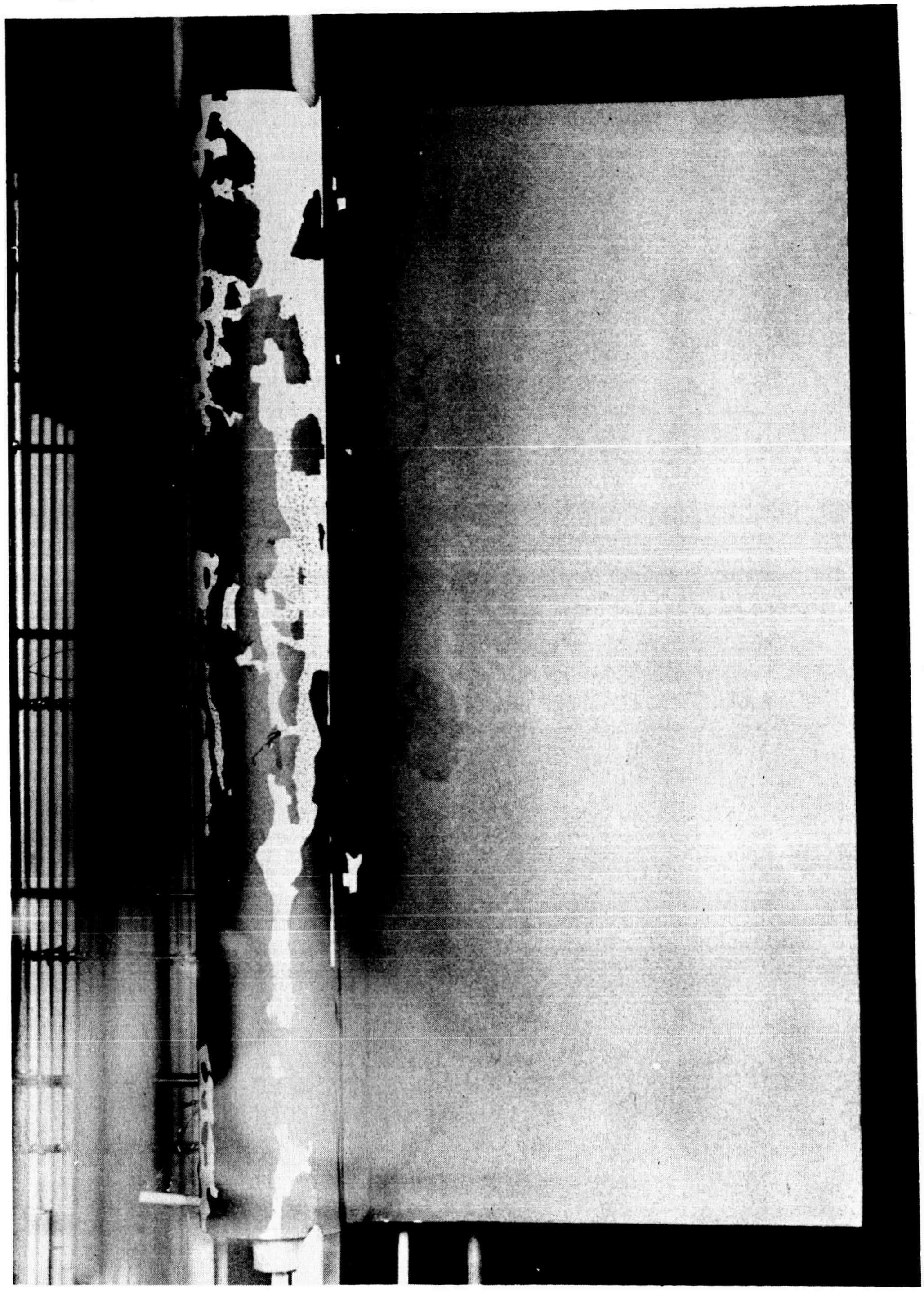
Titania Base Coated SNAP-8 Test Section After 2810 Hours Showing Cracks in Coating on Tube Portion of Specimen

Figure 43



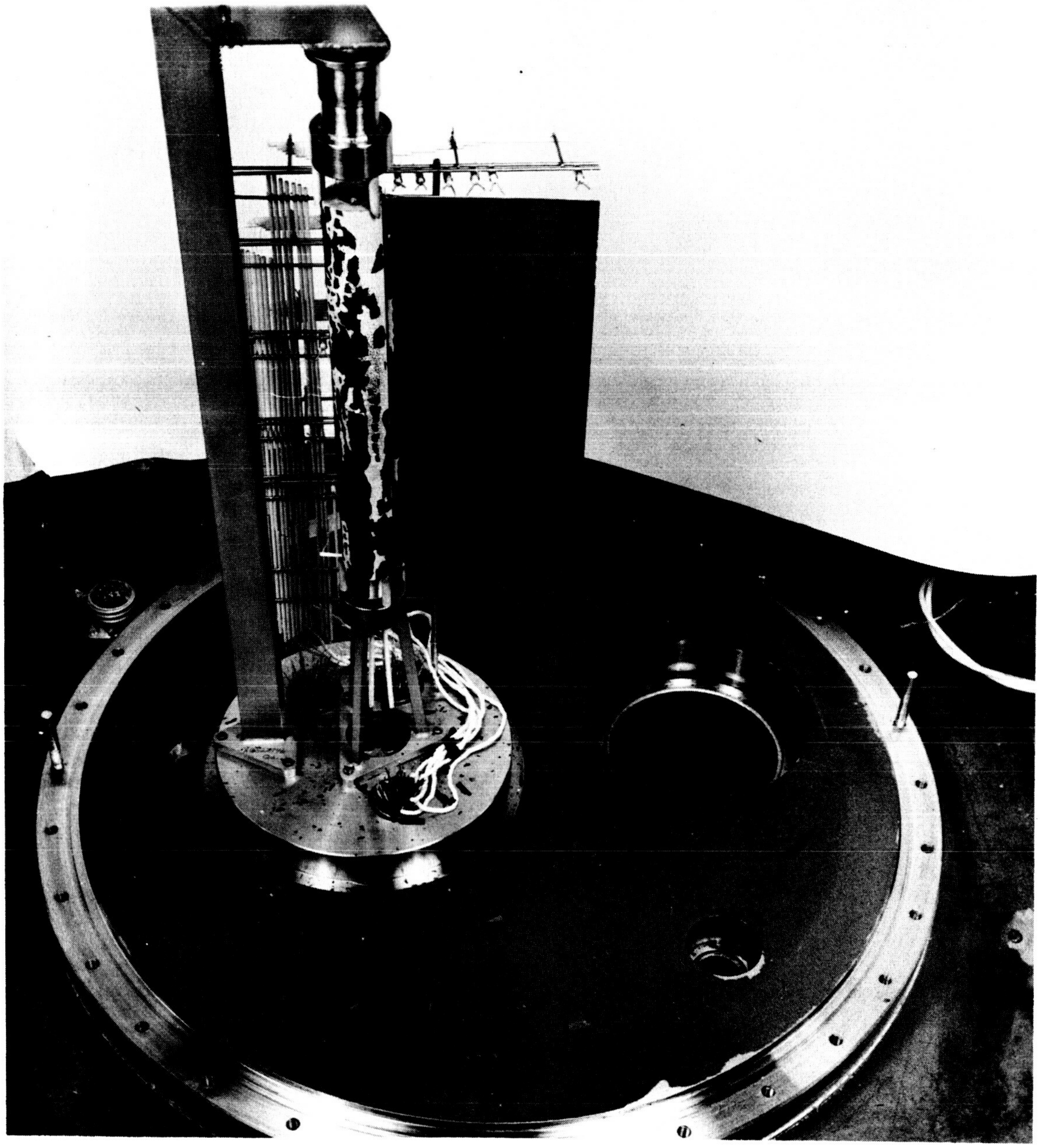
Titania Base Coated SNAP-8 Test Section After 6840 Hours Showing Spalling of Coating on Tube Portion of Specimen

Figure 44



Titania Base Coated SNAP-8 Test Section After Endurance Testing

Figure 45

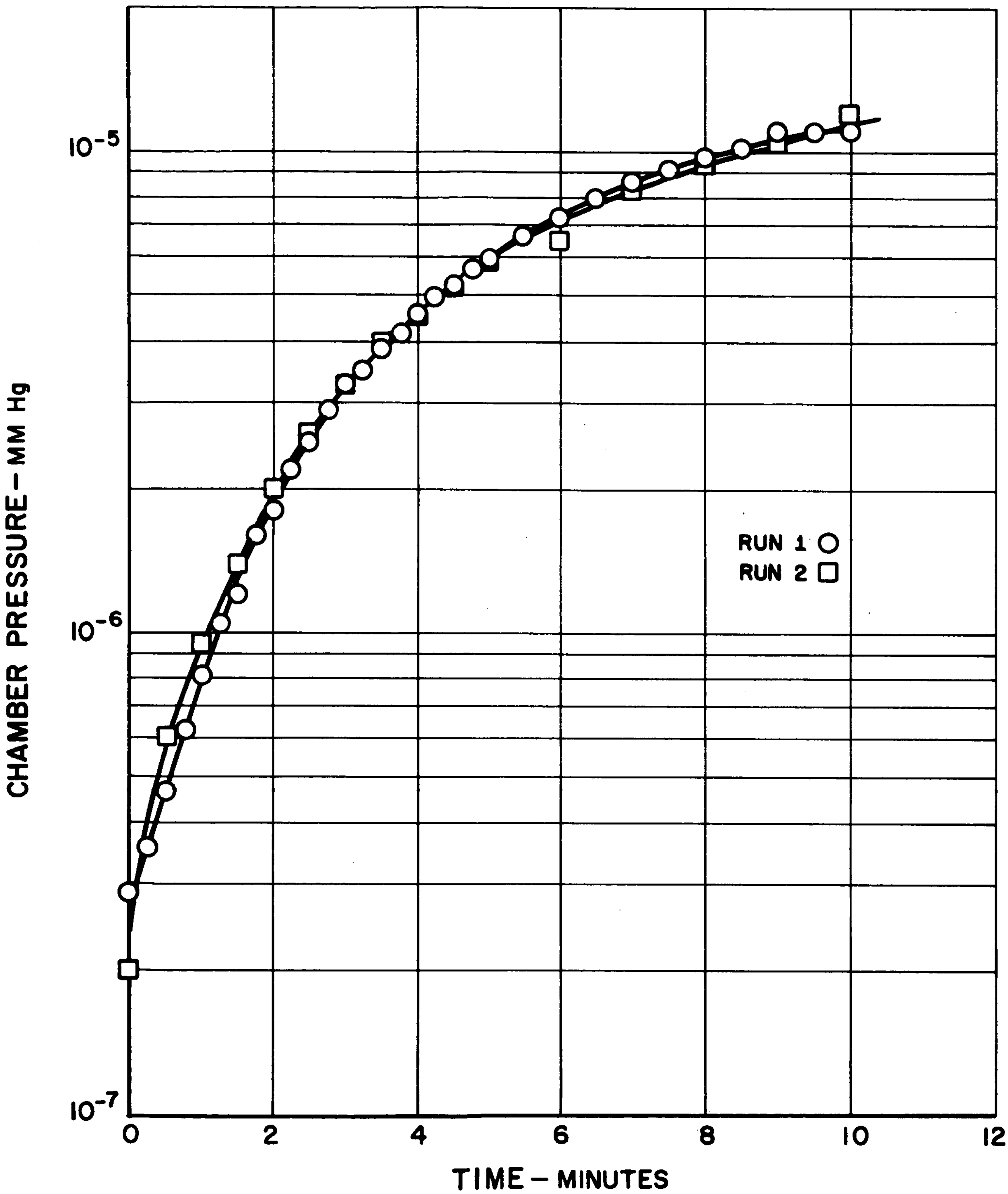


Titania Base Coated SNAP-8 Test Section After Endurance Testing

Figure 46

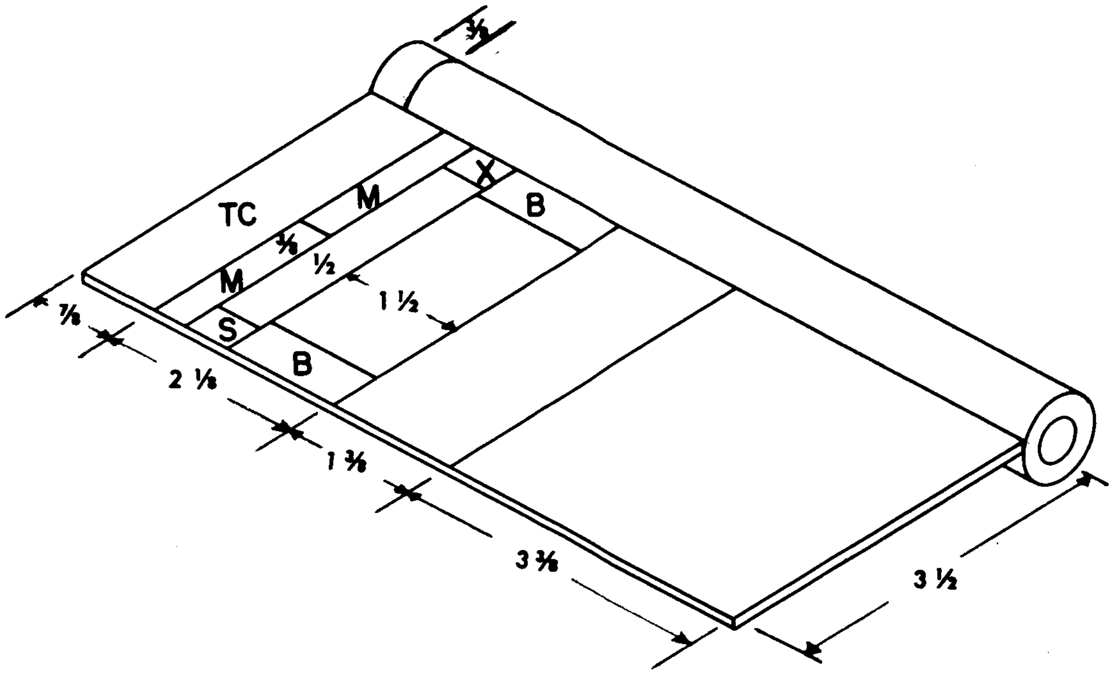
Close-Up View of Tube Portion of Titania Base Coated SNAP-8 Test Section After Endurance Testing

Figure 47



Leak Rate of Test Chamber 2

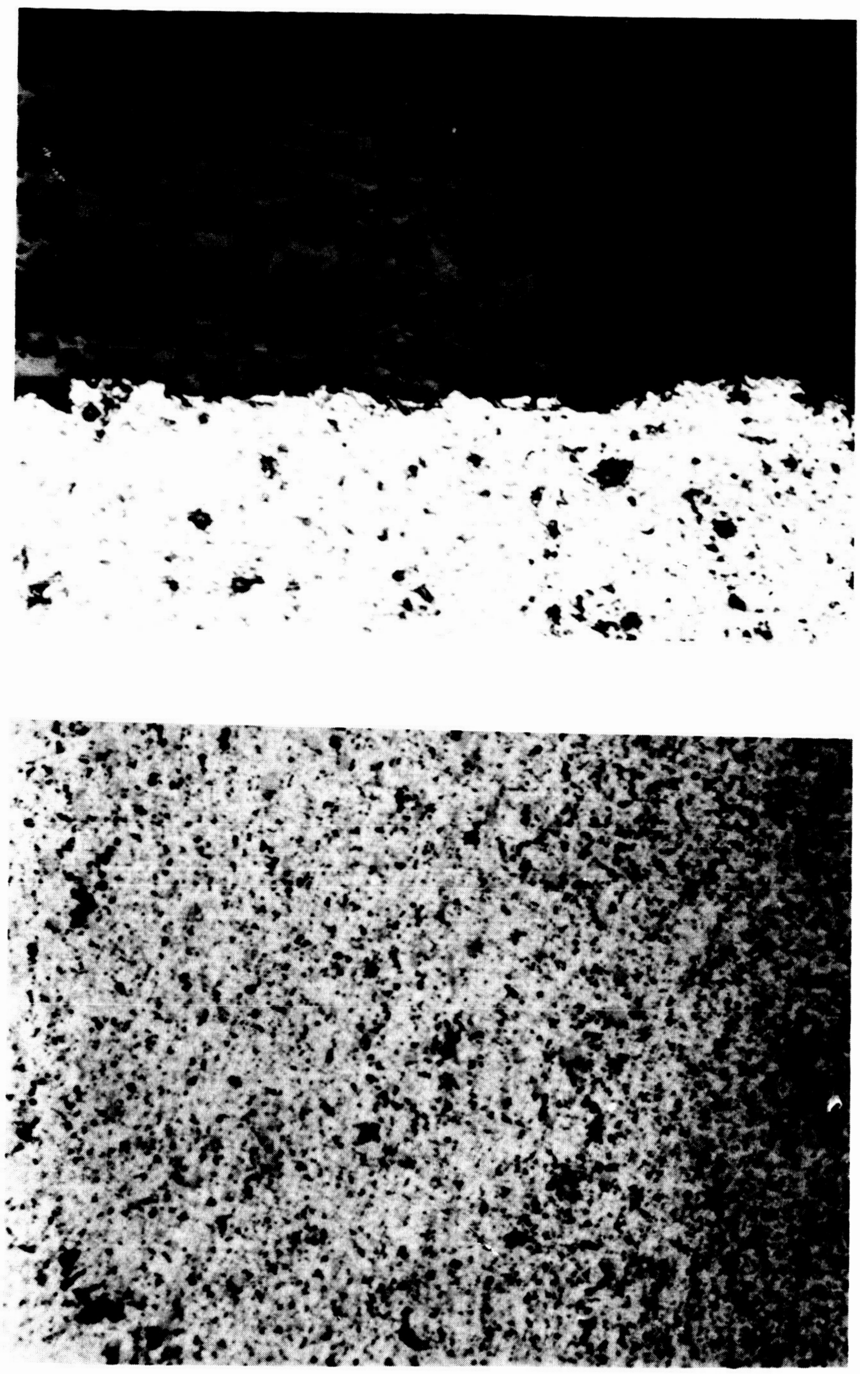
Figure 48



S - SPECTROGRAPHIC ANALYSIS
B - BEND & BOND TESTS
M - METALLOGRAPHIC ANALYSIS
X - X-RAY ANALYSIS
TC - THERMOCOUPLE CHECK

Location of Samples for Post-Test Analyses of Titania Base Coated SNAP-8 Test Section

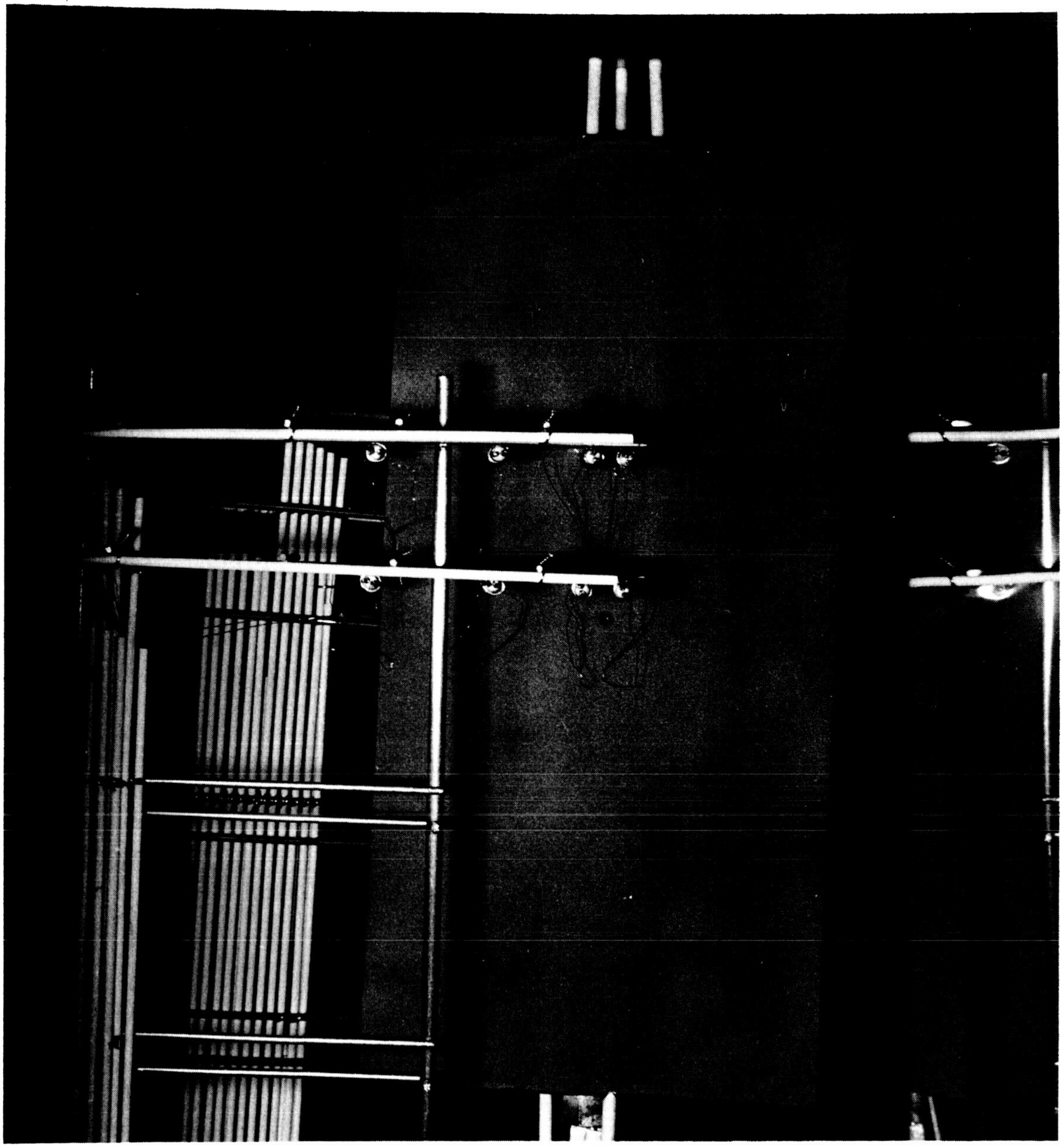
Figure 49



Etchant: 0.5% HF Mag: 500X

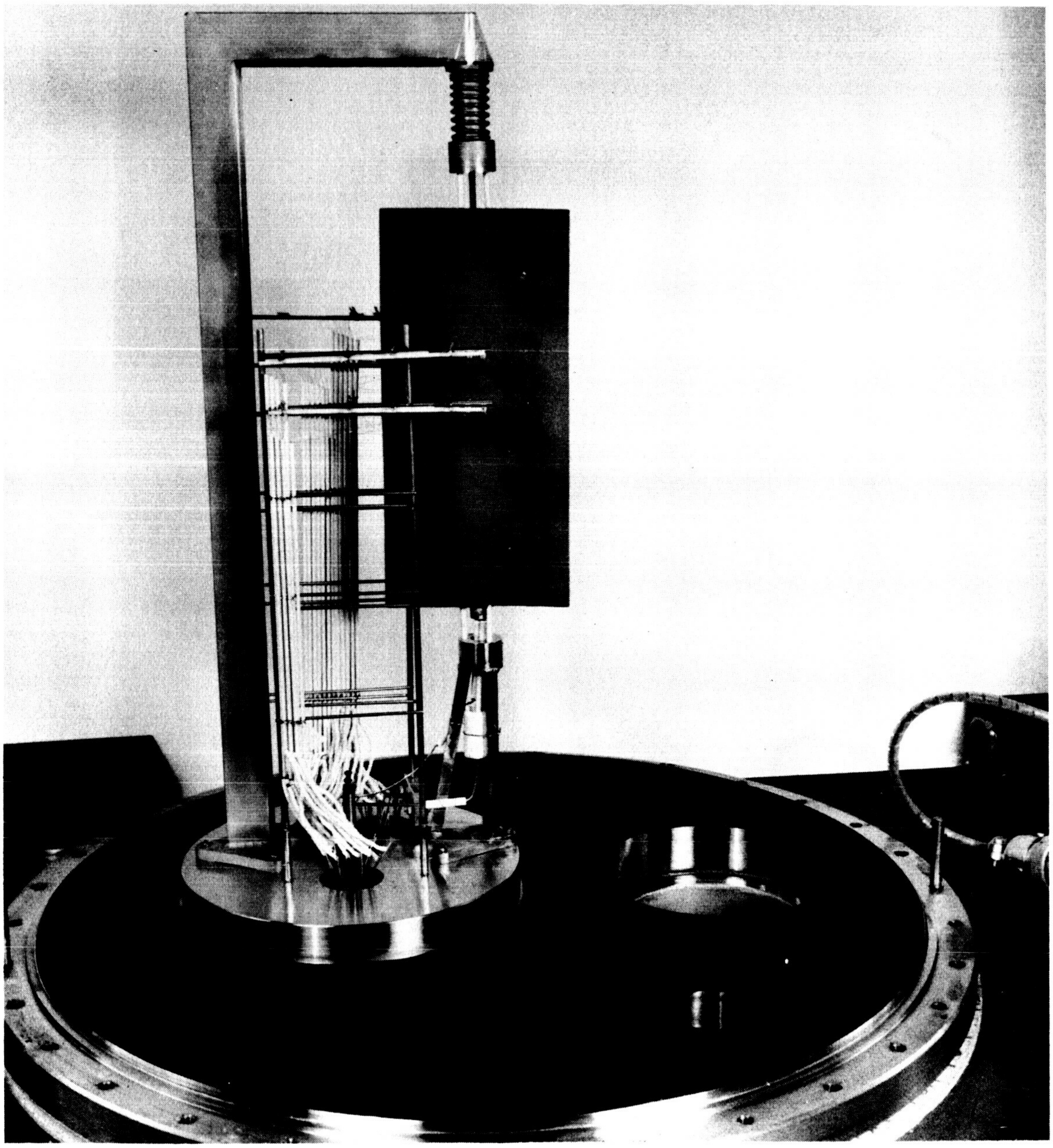
Typical Photomicrographs of Titania Base Coated SNAP-8 Test Section Taken at Locations C (Top) and D (Bottom) Shown in Figure 39.

Figure 50



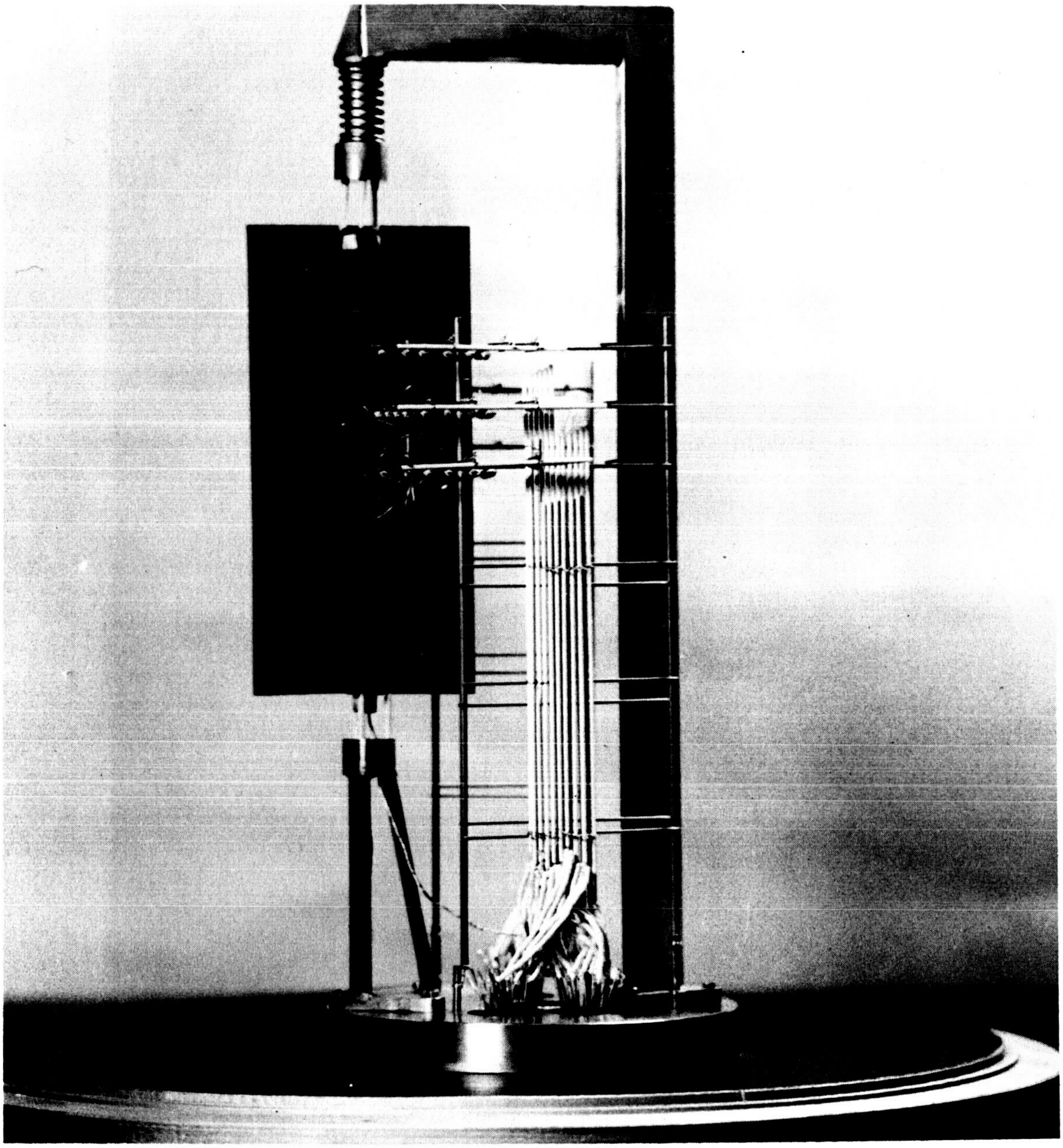
Titania Base Coated Sunflower I Test Section
After Endurance Testing

Figure 51



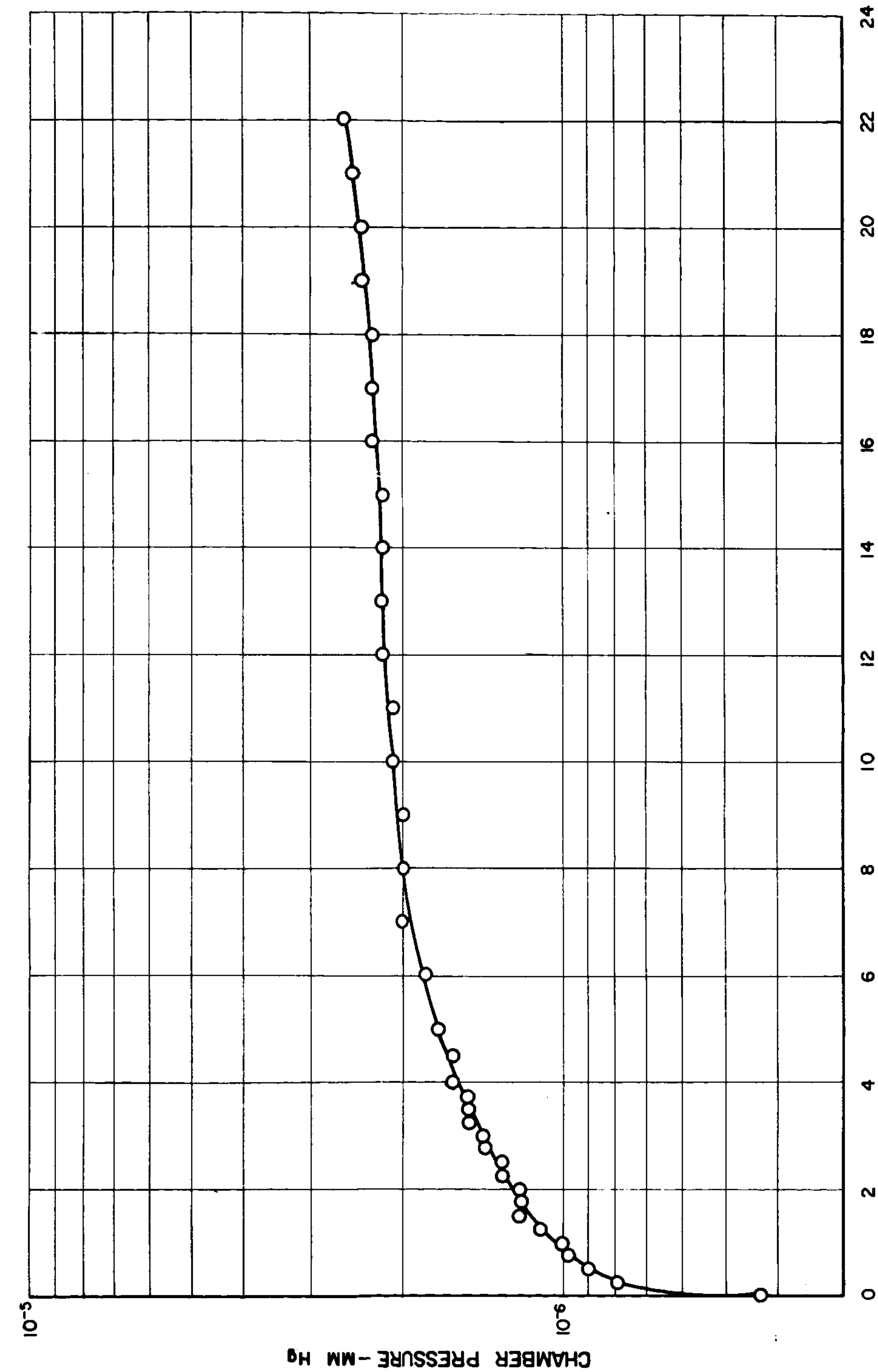
Titania Base Coated Sunflower I Test Section
After Endurance Testing

Figure 52



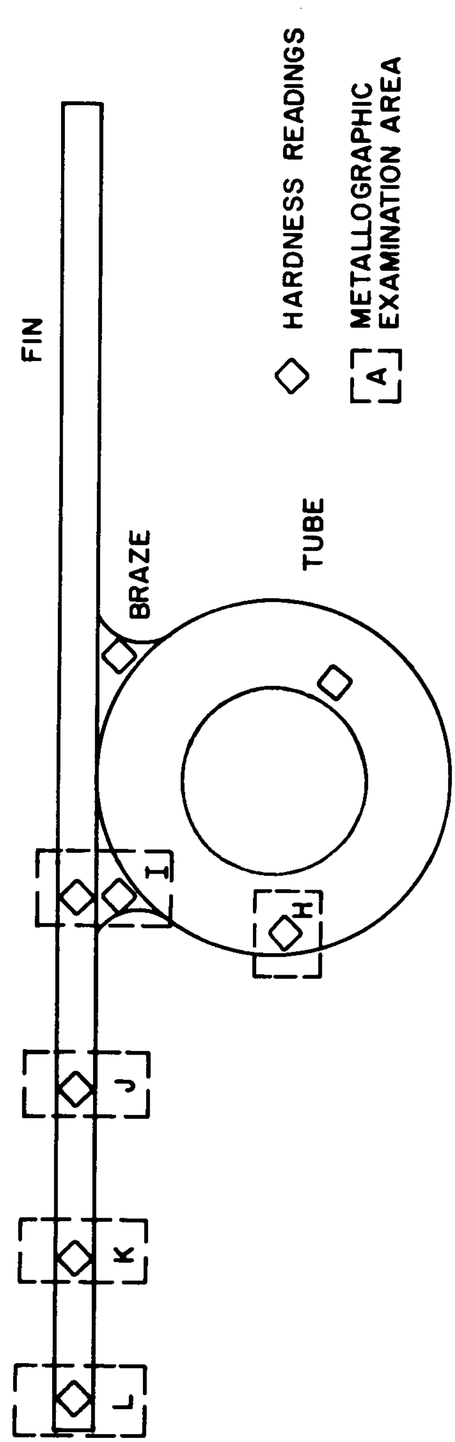
Titania Base Coated Sunflower I Test Section
After Endurance Testing

Figure 53



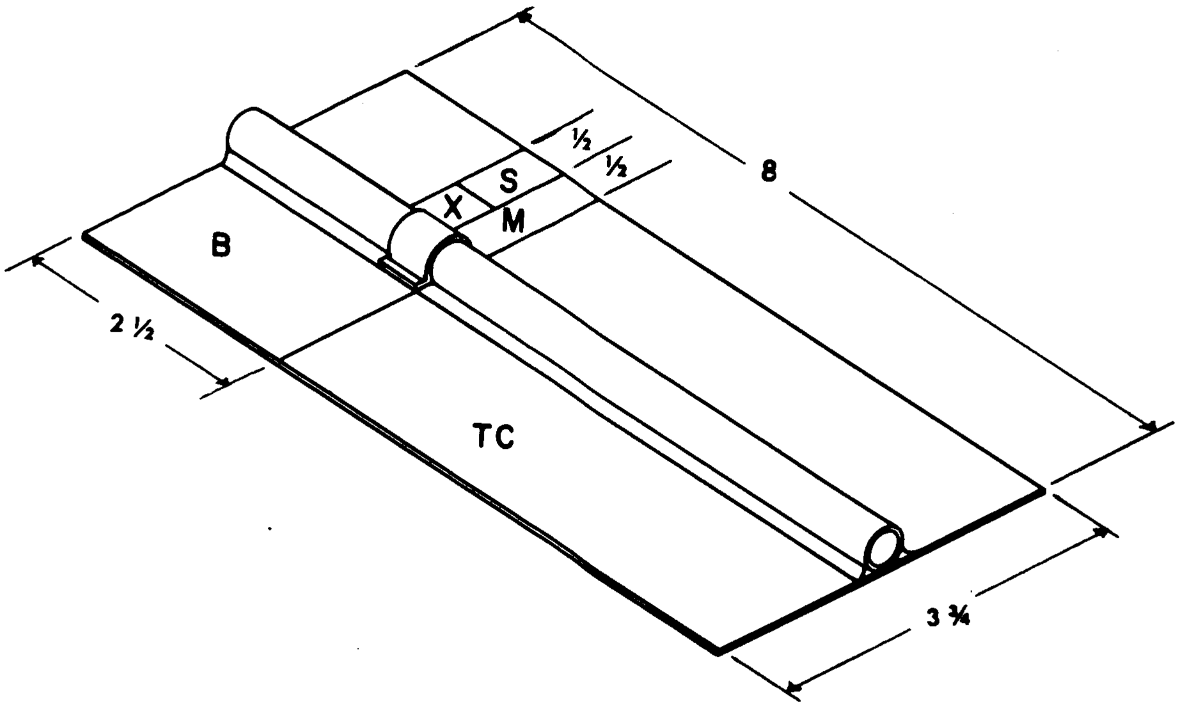
Leak Rate of Test Chamber 3

Figure 54



Cross-Section of Sunflower I Test Section Showing Locations of Hardness Testing and Areas of Metallurgical Examination

Figure 55



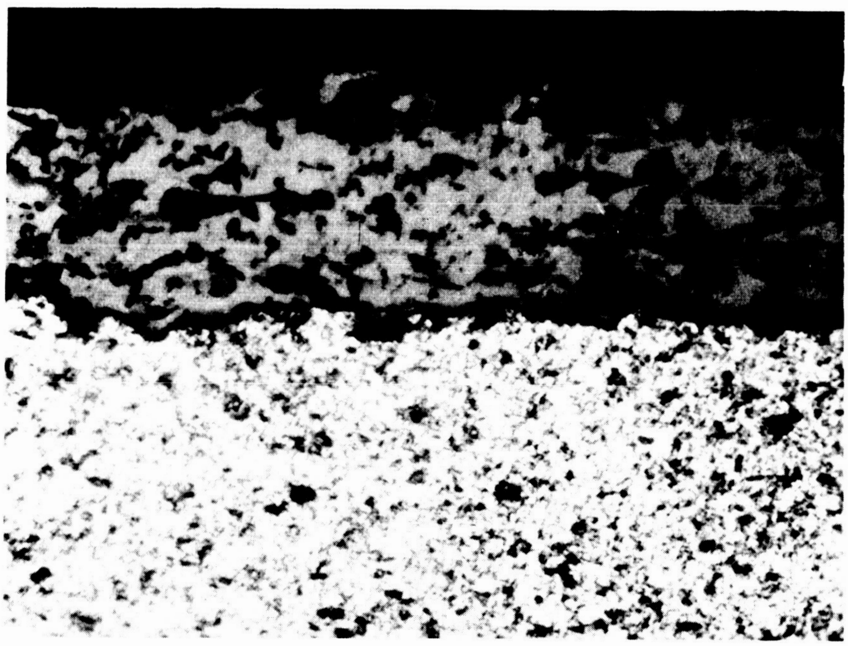
S – SPECTROGRAPHIC ANALYSIS
B – BEND & BOND TESTS
M – METALLOGRAPHIC ANALYSIS
X – X-RAY ANALYSIS
TC – THERMOCOUPLE CHECK

Locations of Samples for Post-Test Analyses of Titania Base Coated Sunflower I Test Section

Figure 56

Coating

Substrate



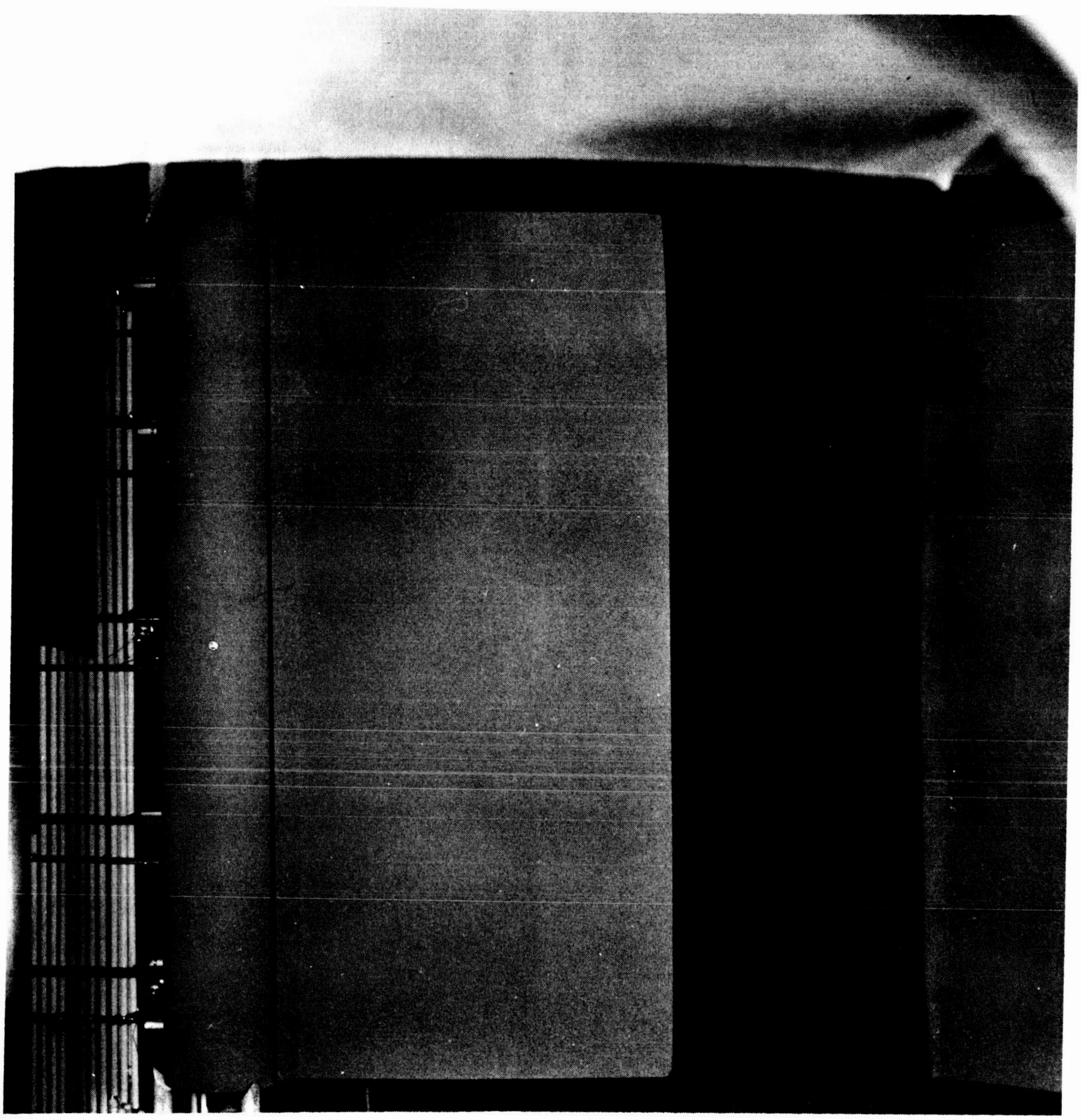
Coating

Substrate

Etchant: 0.5% HF Mag: 500X

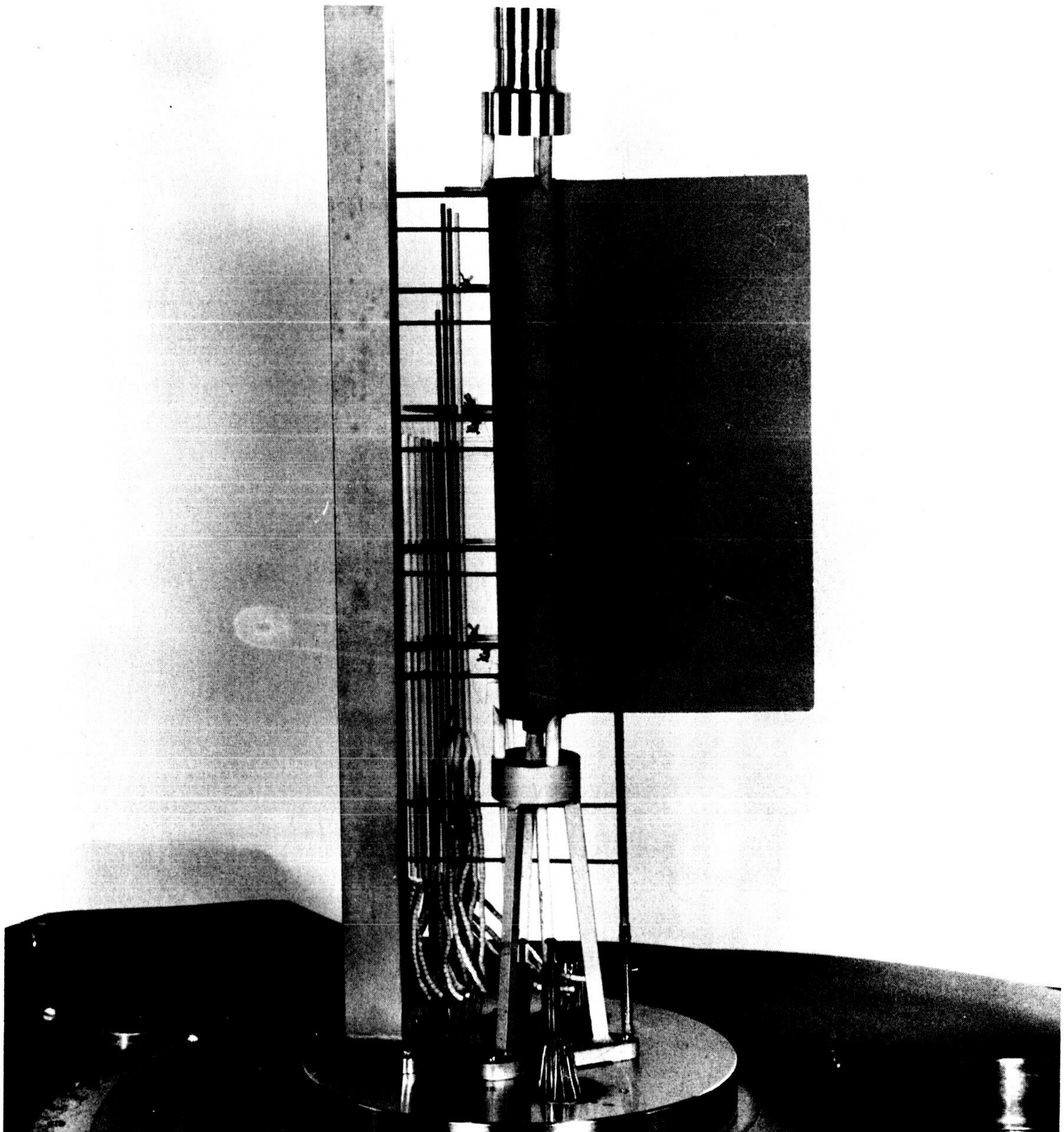
Typical Photomicrographs of Titania Base Coated Sunflower I Test Section Taken at Locations H (Top) and K (Bottom) Shown in Figure 55.

Figure 57



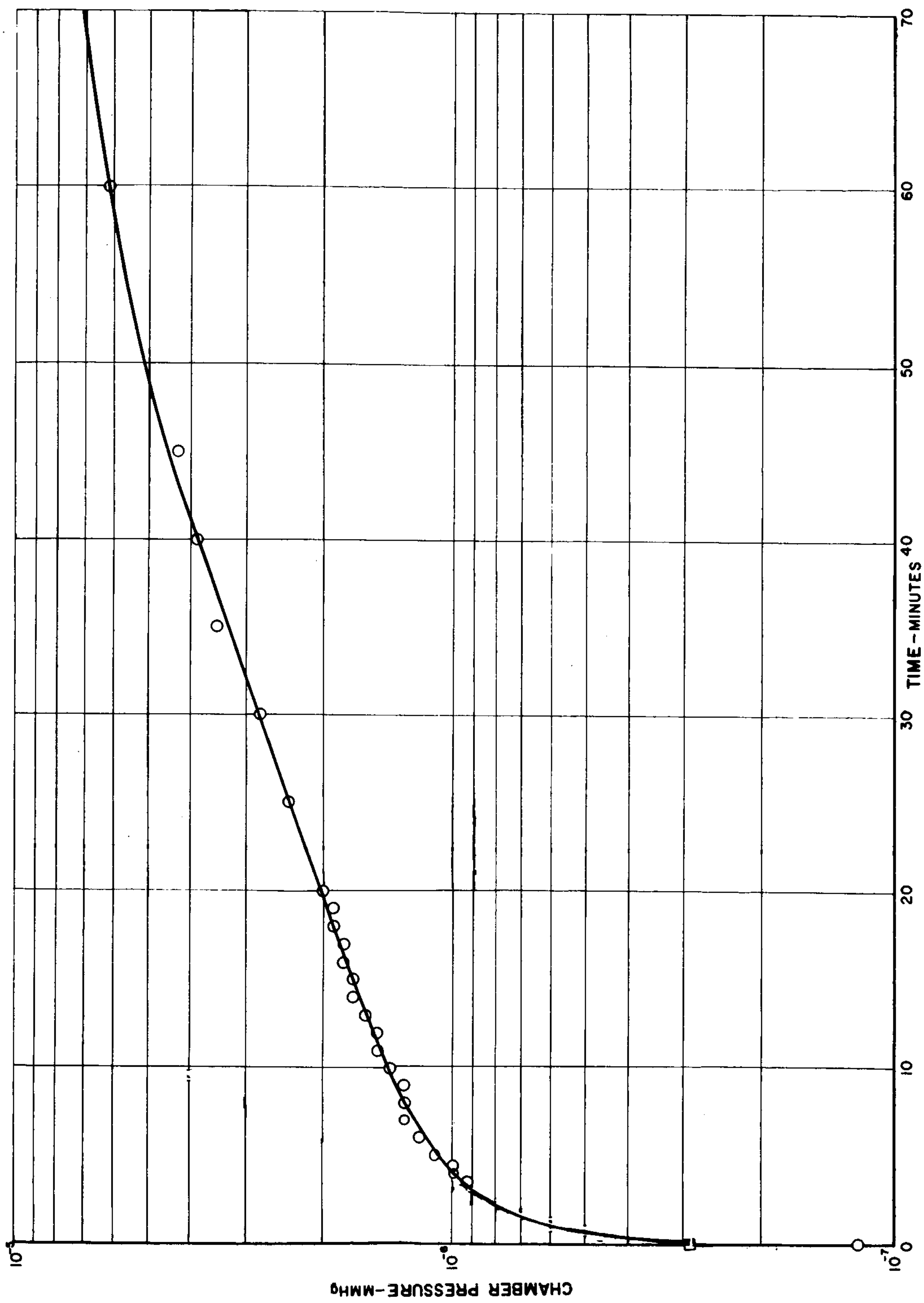
Silicon Carbide and Silicon Dioxide Coated SNAP-8
Test Section After Endurance Testing

Figure 58



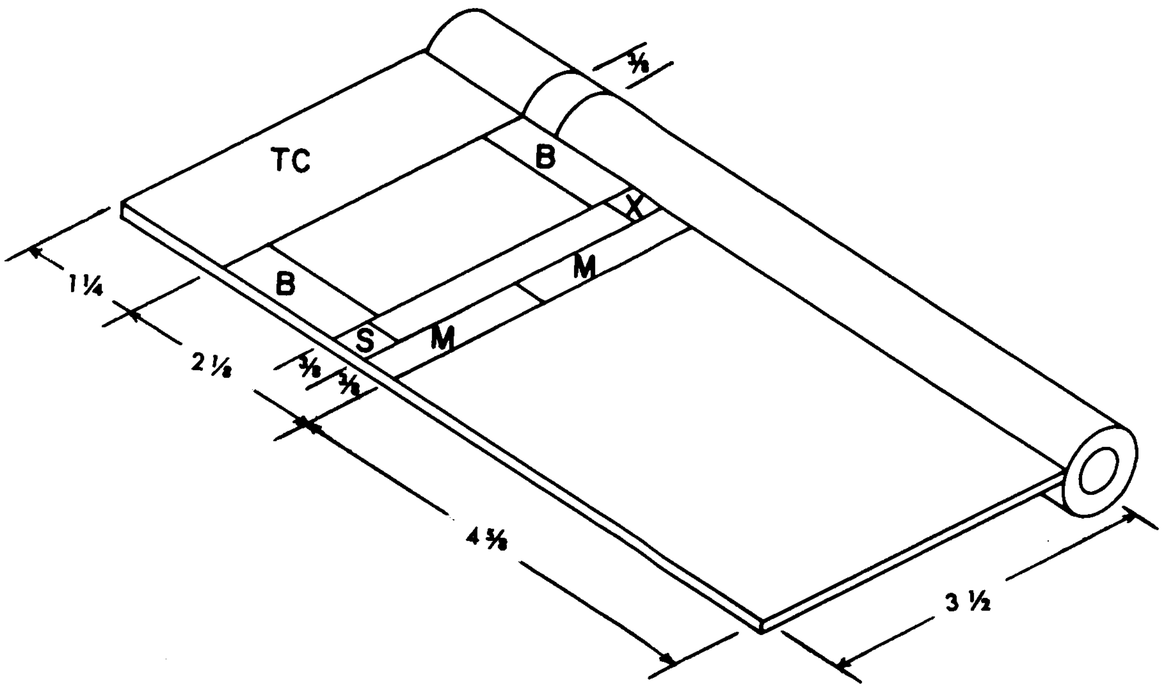
Silicon Carbide and Silicon Dioxide Coated SNAP-8 Test Section After Endurance Testing

Figure 59



Leak Rate of Test Chamber 4

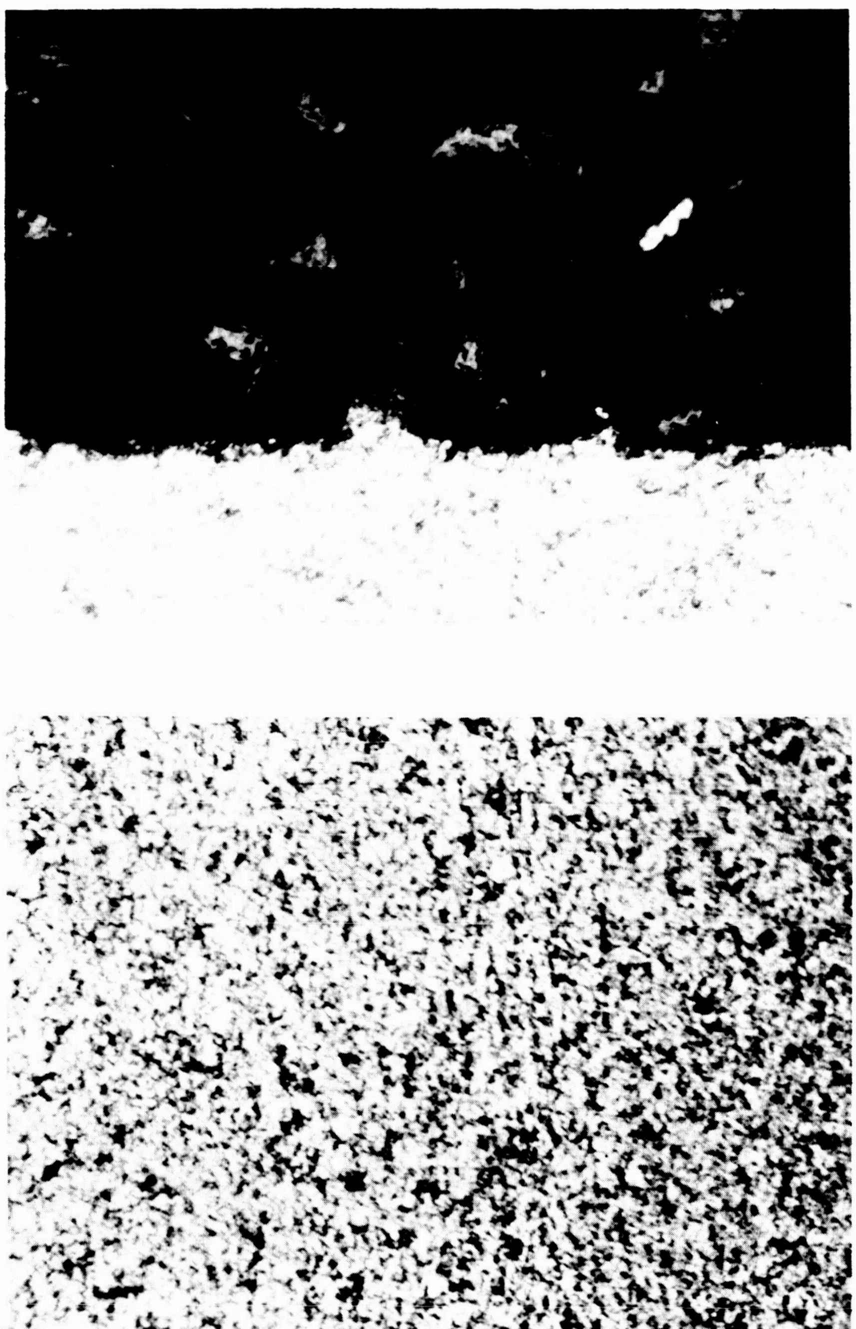
Figure 60



S – SPECTROGRAPHIC ANALYSIS
B – BEND & BOND TESTS
M – METALLOGRAPHIC ANALYSIS
X – X-RAY ANALYSIS
TC – THERMOCOUPLE CHECK

Locations of Samples for Post-Test Analyses of Silicon Carbide and Silicon Dioxide Coated SNAP-8 Test Section

Figure 61



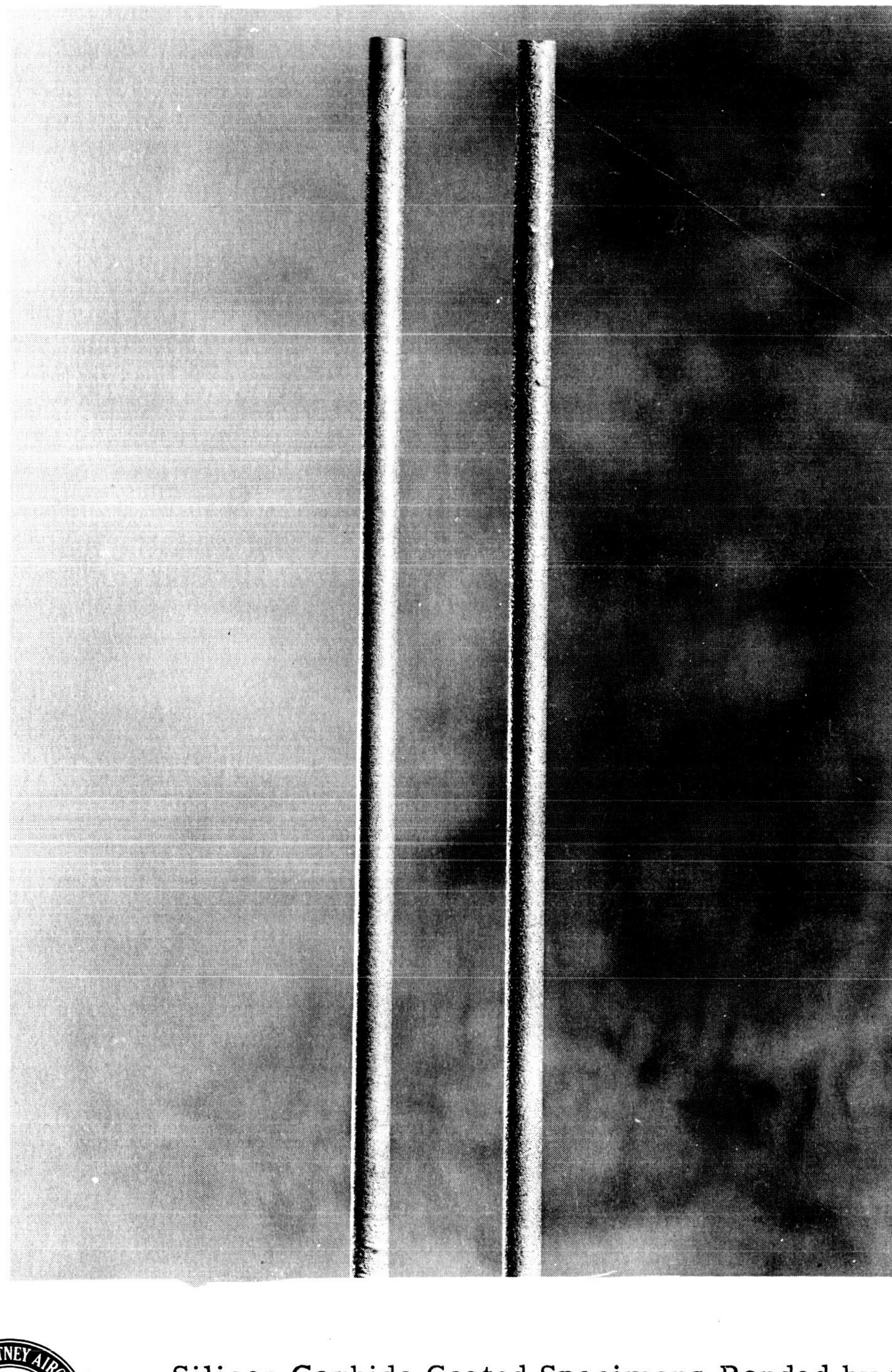
Coating

Substrate

Etchant: 0.5% HF Mag: 500X

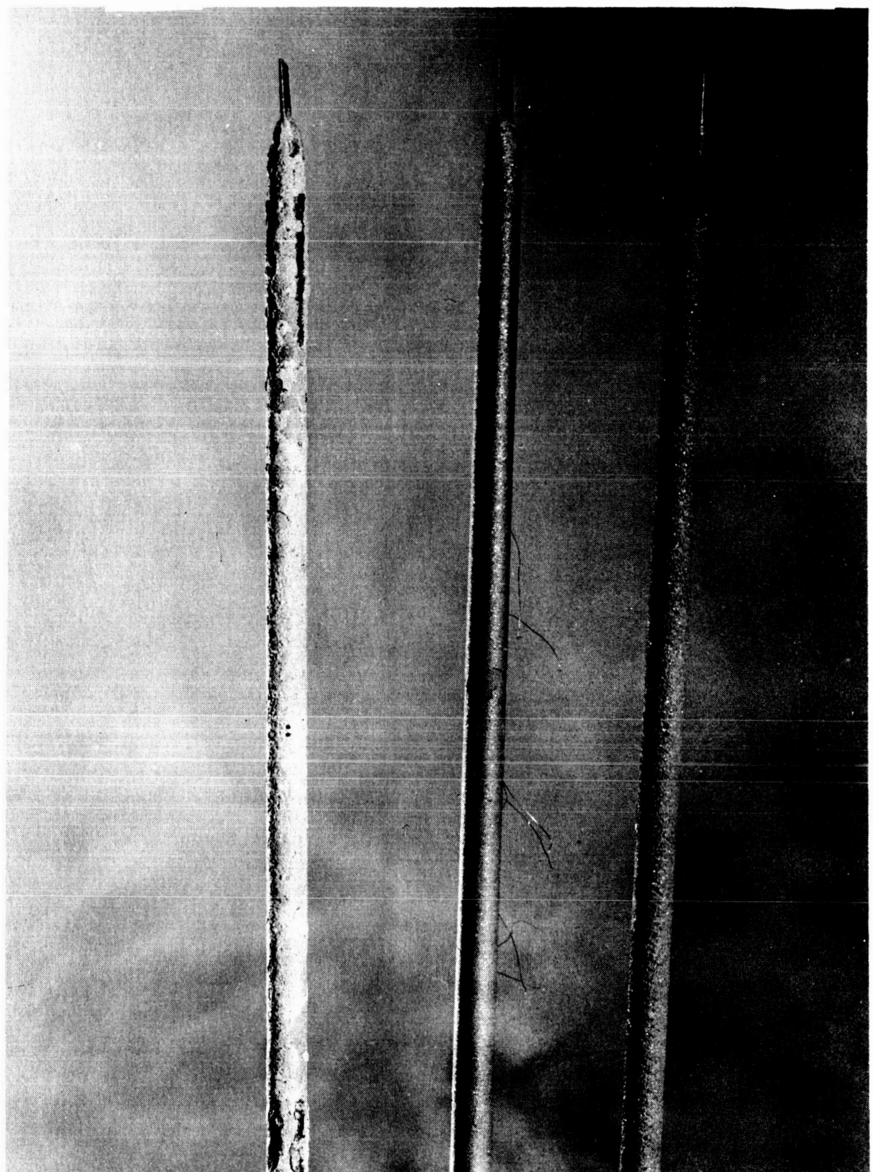
Typical Photomicrographs of Silicon Carbide and Silicon Dioxide Coated SNAP-8 Test Section Taken at Locations C (Top) and D (Bottom) Shown in Figure 39.

Figure 62



Silicon Carbide Coated Specimens Bonded by the Original (Left) and the Modified (Right) Method

Figure 63



Silicon Carbide Coated Specimens Bonded by the Original (Left) and Modified (Center and Right) Method After Testing

Figure 64

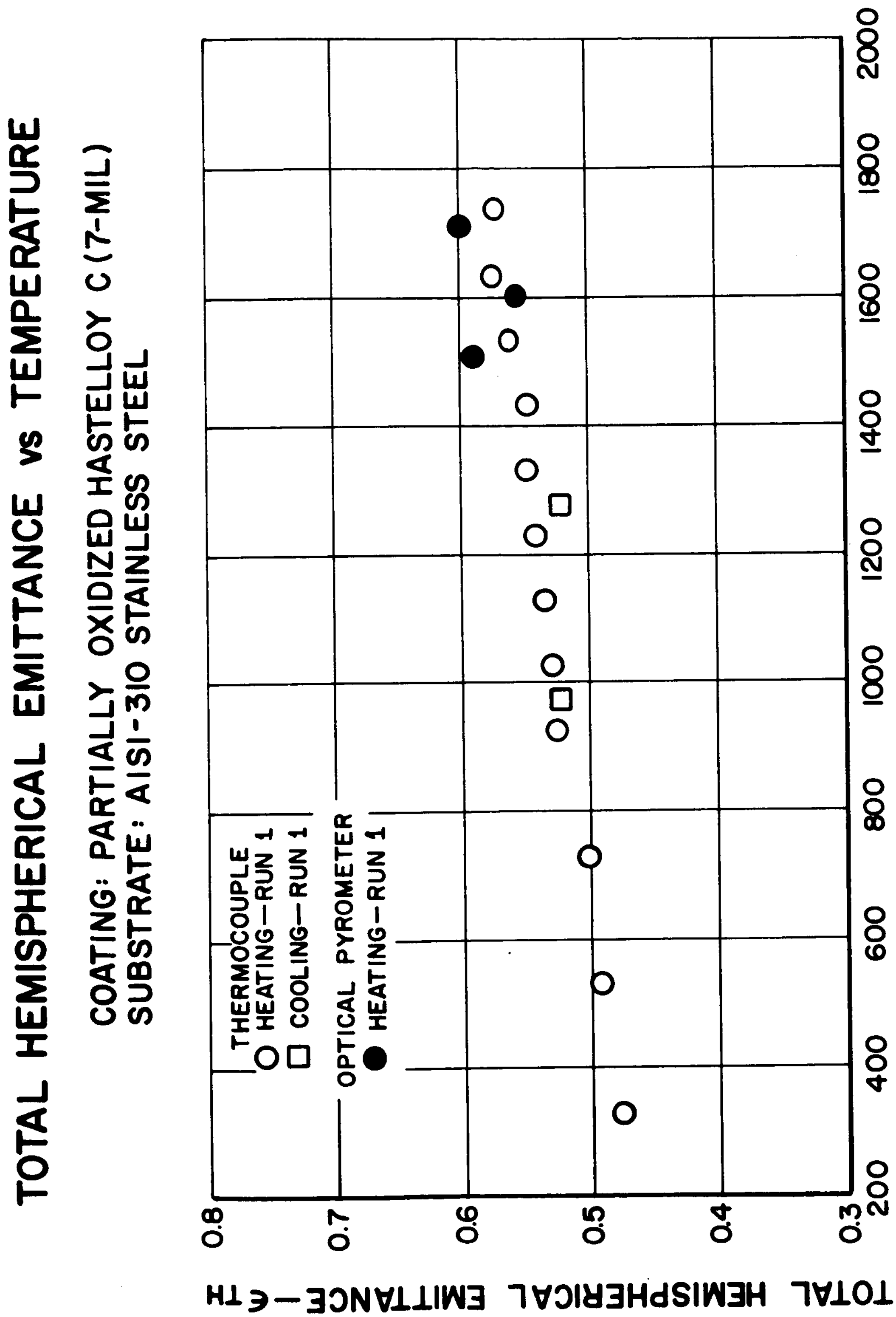

TOTAL HEMISPHERICAL EMITTANCE vs TEMPERATURE
COATING: PARTIALLY OXIDIZED HASTELLOY C (7-MIL)
SUBSTRATE: AISI-310 STAINLESS STEEL
THERMOCOUPLE
HEATING—RUN 1
COOLING—RUN 1
OPTICAL PYROMETER
HEATING—RUN 1
0.8
0.7
0.6
0.5
0.4
0.3
TOTAL HEMISPHERICAL EMITTANCE—ϵTH
200
400
600
800
1000
1200
1400
1600
1800
2000
TEMPERATURE—°F

Figure 65

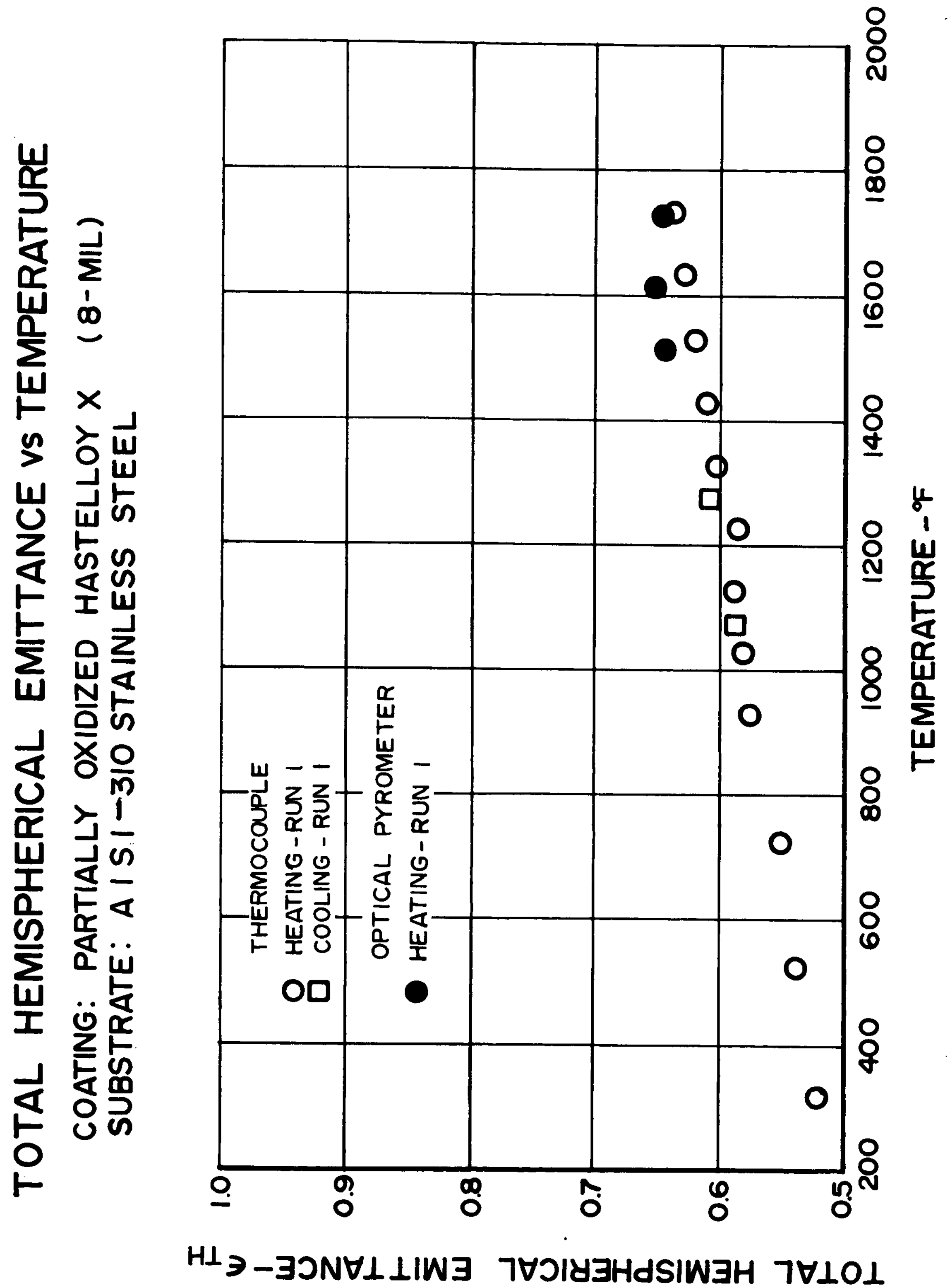

TOTAL HEMISPHERICAL EMITTANCE vs TEMPERATURE
COATING: PARTIALLY OXIDIZED HASTELLOY X (8-MIL)
SUBSTRATE: A I S I -310 STAINLESS STEEL
THERMOCOUPLE
HEATING-RUN I
COOLING-RUN I
OPTICAL PYROMETER
HEATING-RUN I
TOTAL HEMISPHERICAL EMITTANCE - ϵ_{TH}
1.0
0.9
0.8
0.7
0.6
0.5
200
400
600
800
1000
1200
1400
1600
1800
2000
TEMPERATURE - °F

Figure 66

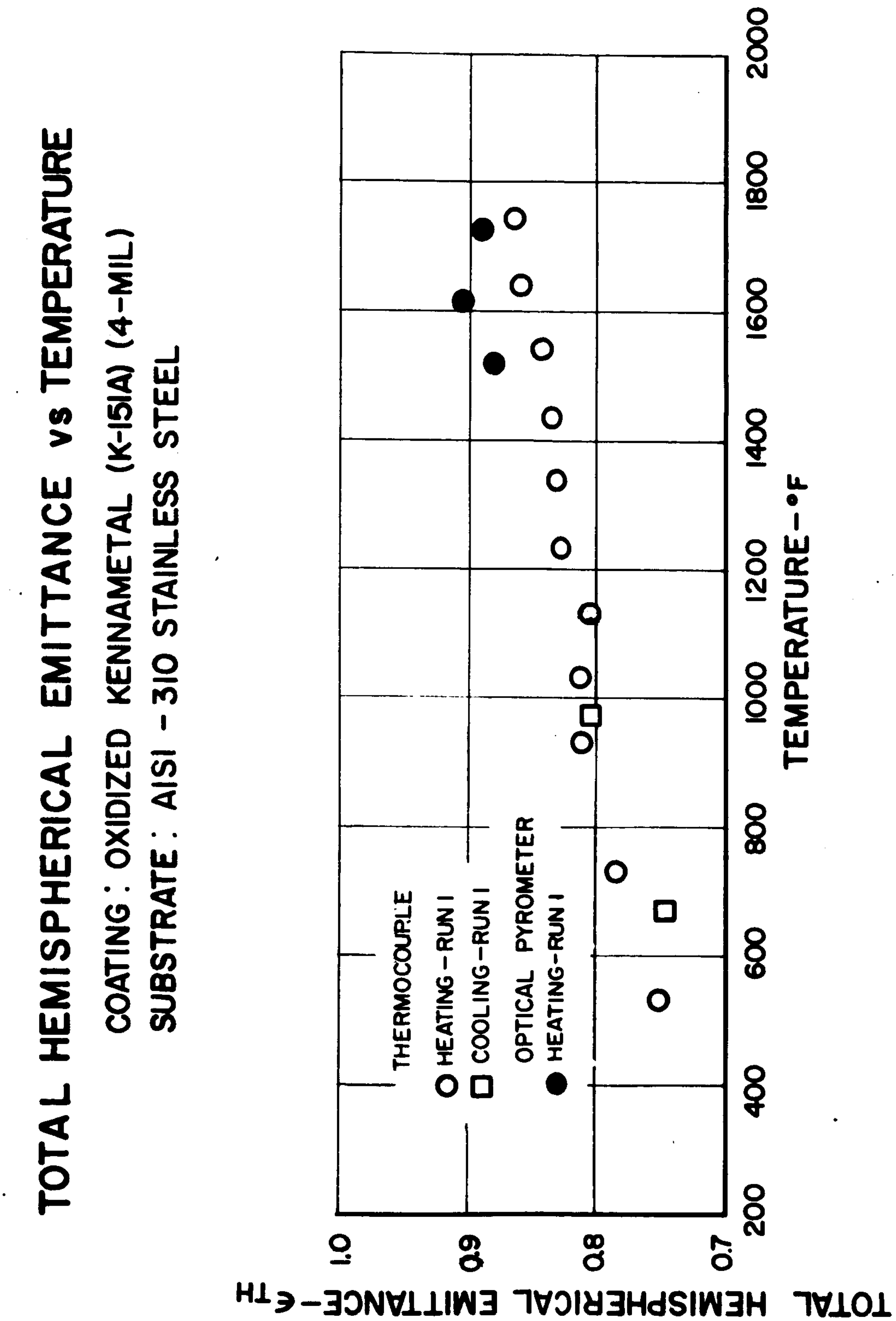

TOTAL HEMISPHERICAL EMITTANCE vs TEMPERATURE
COATING: OXIDIZED KENNAMETAL (K-151A) (4-MIL)
SUBSTRATE: AISI - 310 STAINLESS STEEL
THERMOCOUPLE
HEATING-RUN I
COOLING-RUN I
OPTICAL PYROMETER
HEATING-RUN I
1.0
0.9
0.8
0.7
200
400
600
800
1000
1200
1400
1600
1800
2000
TEMPERATURE-°F
TOTAL HEMISPHERICAL EMITTANCE-ϵ_{TH}

Figure 67

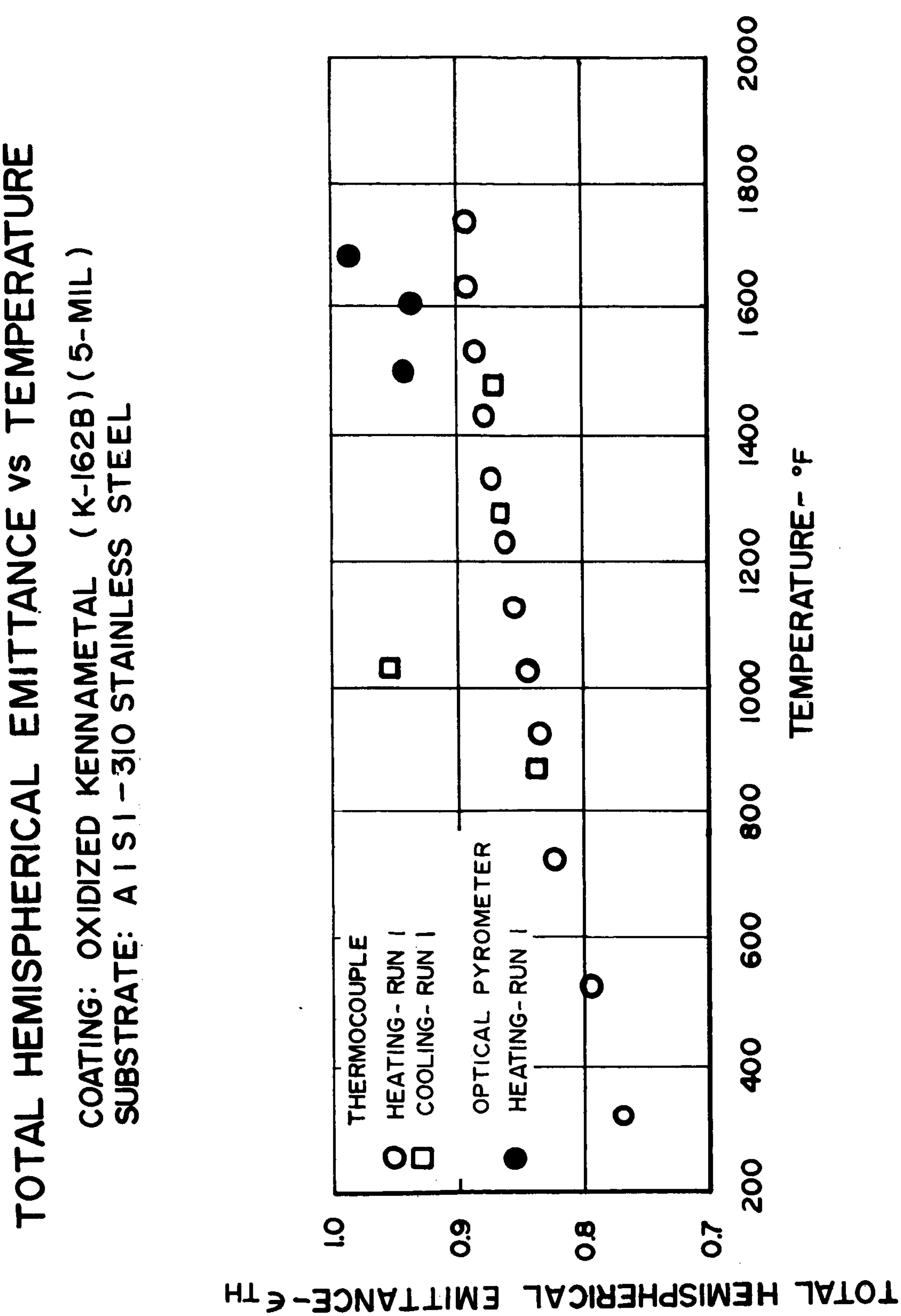

TOTAL HEMISPHERICAL EMITTANCE vs TEMPERATURE
COATING: OXIDIZED KENNAMETAL (K-162B)(5-MIL)
SUBSTRATE: AISI-310 STAINLESS STEEL
THERMOCOUPLE
HEATING-RUN I
COOLING-RUN I
OPTICAL PYROMETER
HEATING-RUN I
TOTAL HEMISPHERICAL EMITTANCE-ϵ_{TH}
1.0
0.9
0.8
0.7
200
400
600
800
1000
1200
1400
1600
1800
2000
TEMPERATURE-°F

Figure 68

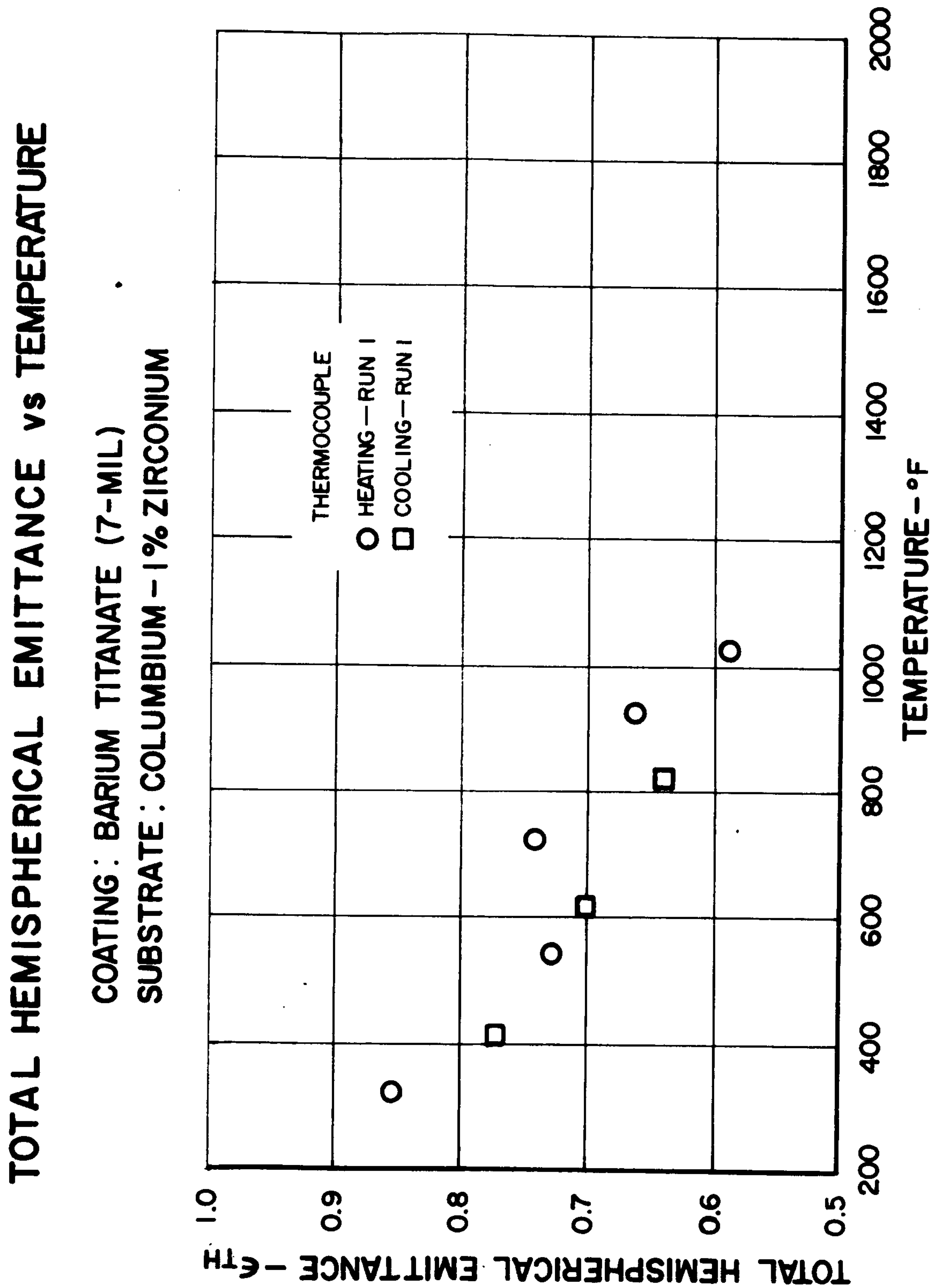

TOTAL HEMISPHERICAL EMITTANCE vs TEMPERATURE
COATING: BARIUM TITANATE (7-MIL)
SUBSTRATE: COLUMBIUM - 1% ZIRCONIUM
THERMOCOUPLE
HEATING—RUN I
COOLING—RUN I
TOTAL HEMISPHERICAL EMITTANCE — ϵ_{TH}
1.0
0.9
0.8
0.7
0.6
0.5
200
400
600
800
1000
1200
1400
1600
1800
2000
TEMPERATURE — °F

Figure 69

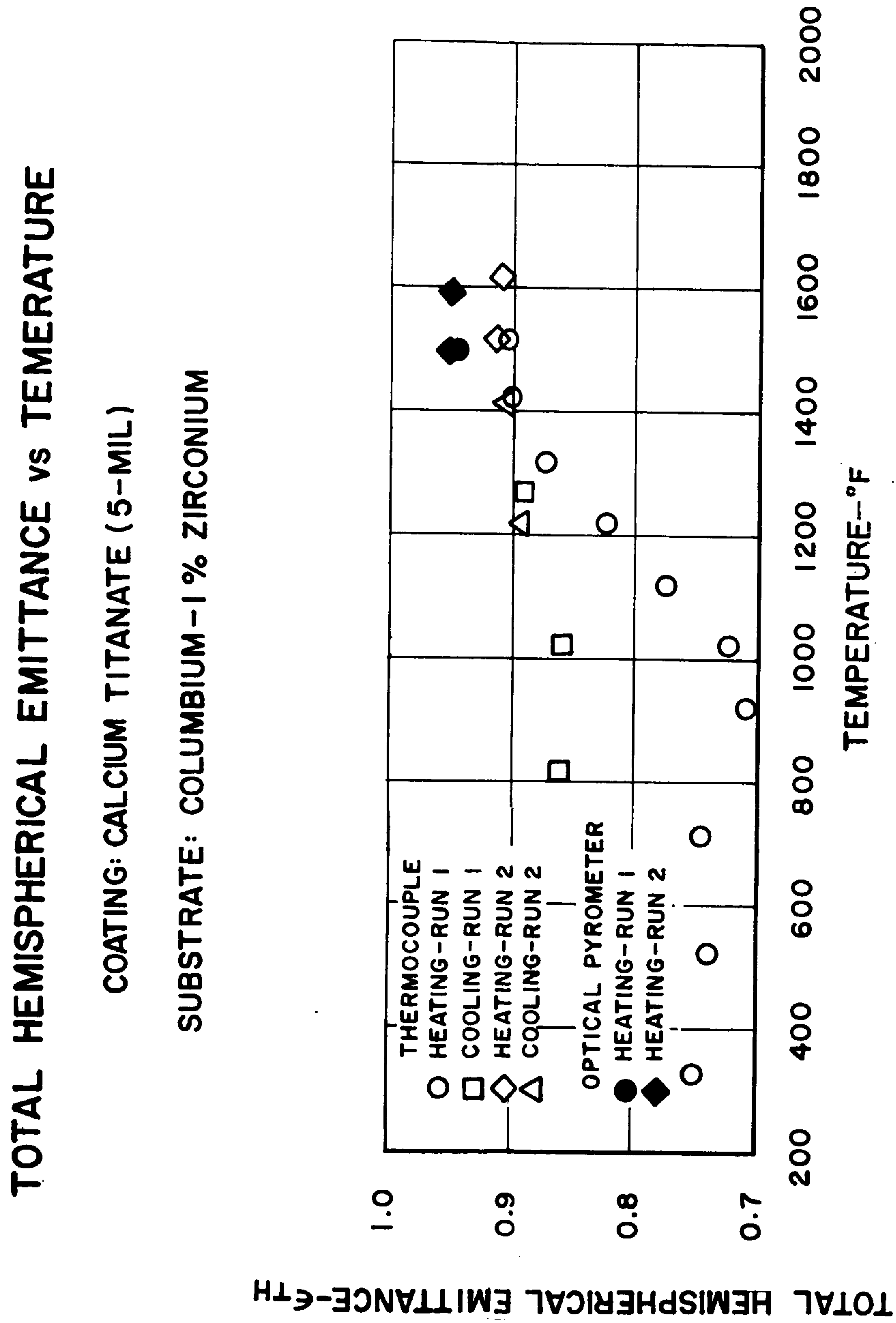

TOTAL HEMISPHERICAL EMITTANCE vs TEMERATURE
COATING: CALCIUM TITANATE (5-MIL)
SUBSTRATE: COLUMBIUM-1 % ZIRCONIUM
THERMOCOUPLE
HEATING-RUN 1
COOLING-RUN 1
HEATING-RUN 2
COOLING-RUN 2
OPTICAL PYROMETER
HEATING-RUN 1
HEATING-RUN 2
1.0
0.9
0.8
0.7
200
400
600
800
1000
1200
1400
1600
1800
2000
TEMPERATURE--°F
TOTAL HEMISPHERICAL EMITTANCE-ϵTH

Figure 70

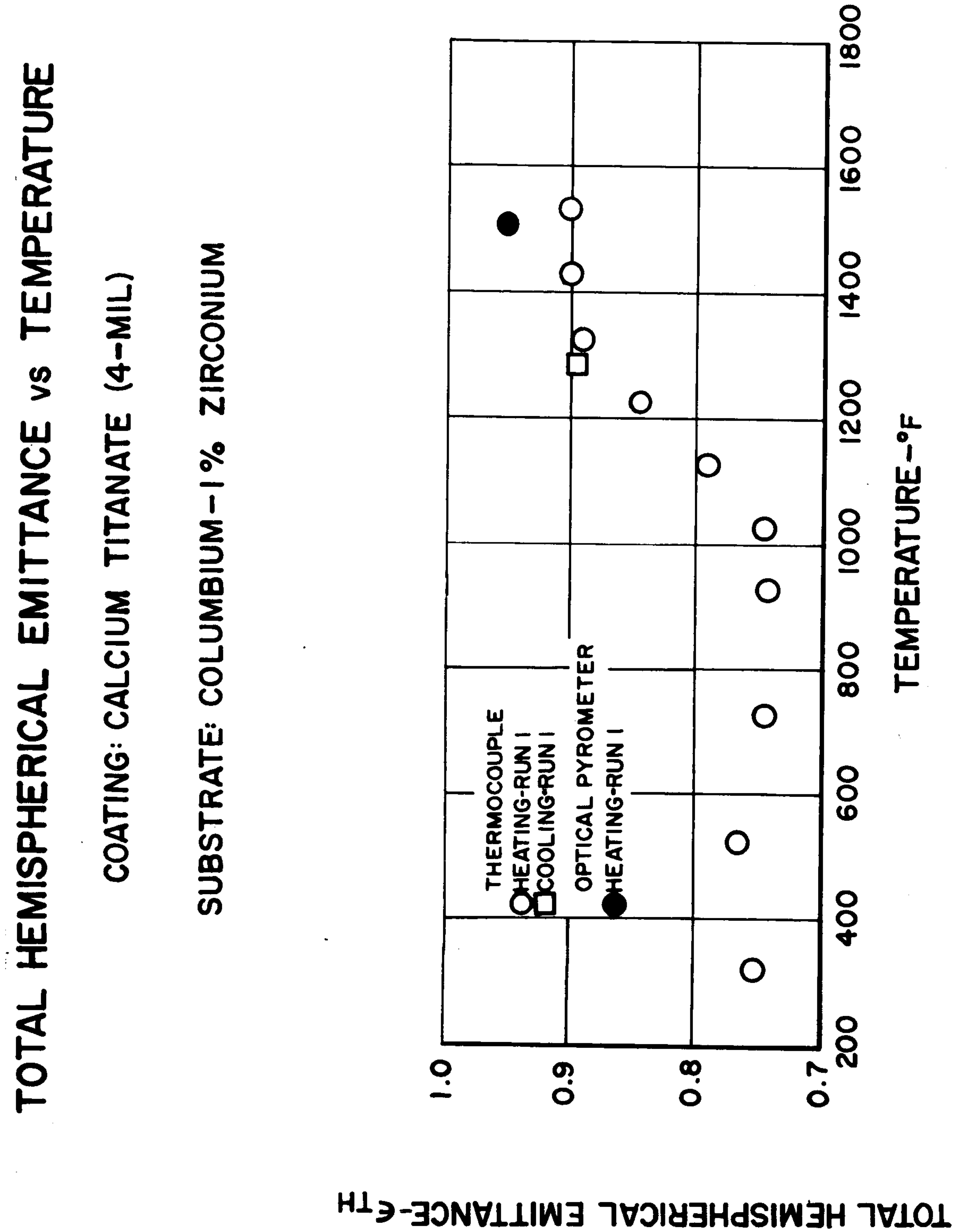

TOTAL HEMISPHERICAL EMITTANCE vs TEMPERATURE
COATING: CALCIUM TITANATE (4-MIL)
SUBSTRATE: COLUMBIUM-1% ZIRCONIUM
THERMOCOUPLE
HEATING-RUN I
COOLING-RUN I
OPTICAL PYROMETER
HEATING-RUN I
1.0
0.9
0.8
0.7
200
400
600
800
1000
1200
1400
1600
1800
TEMPERATURE-°F
TOTAL HEMISPHERICAL EMITTANCE-ϵ_{TH}

Figure 71

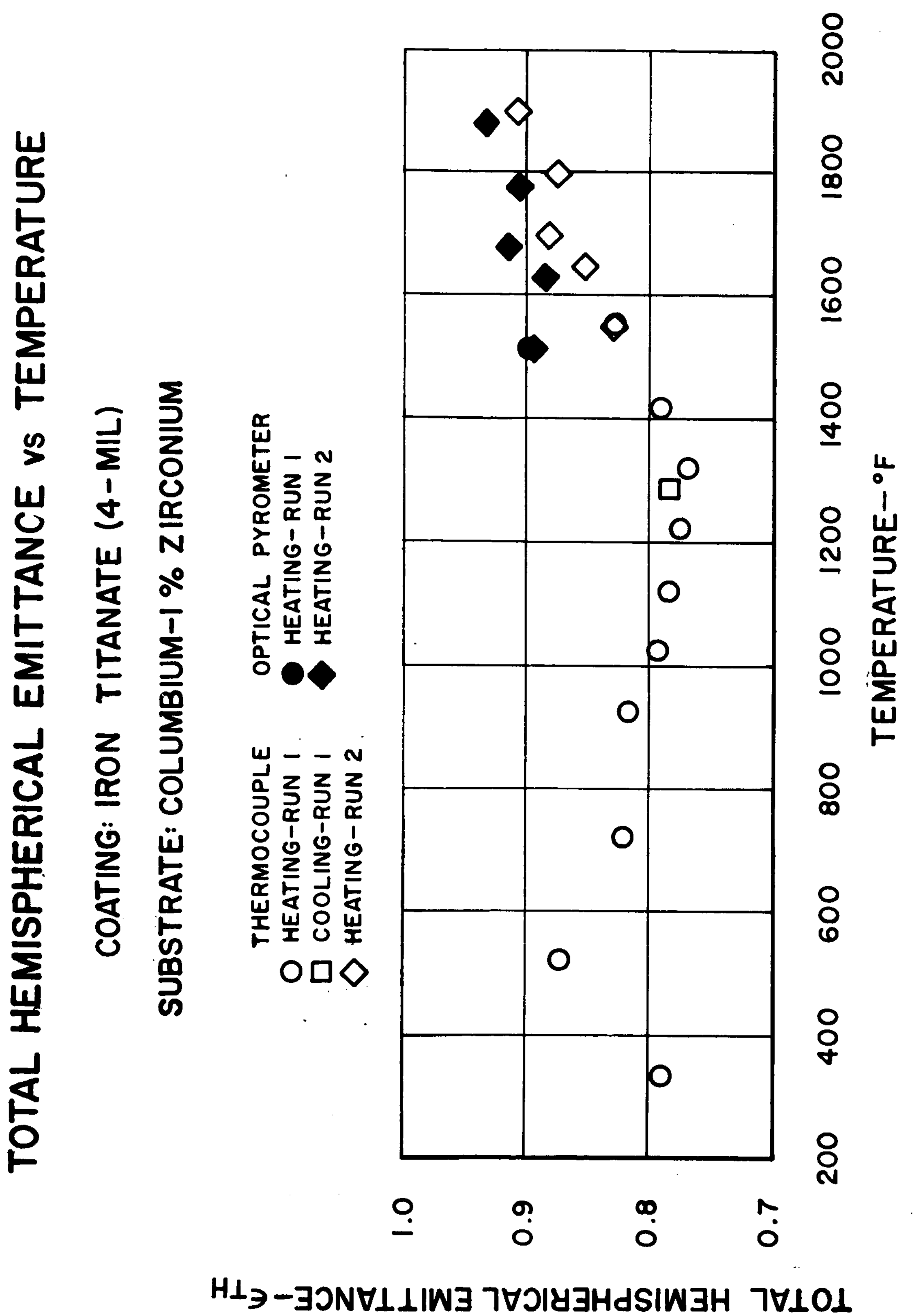

TOTAL HEMISPHERICAL EMITTANCE vs TEMPERATURE
COATING: IRON TITANATE (4-MIL)
SUBSTRATE: COLUMBIUM-1 % ZIRCONIUM
THERMOCOUPLE
HEATING-RUN 1
COOLING-RUN 1
HEATING-RUN 2
OPTICAL PYROMETER
HEATING-RUN 1
HEATING-RUN 2
1.0
0.9
0.8
0.7
TOTAL HEMISPHERICAL EMITTANCE—ϵ_{TH}
200
400
600
800
1000
1200
1400
1600
1800
2000
TEMPERATURE—°F

Figure 72

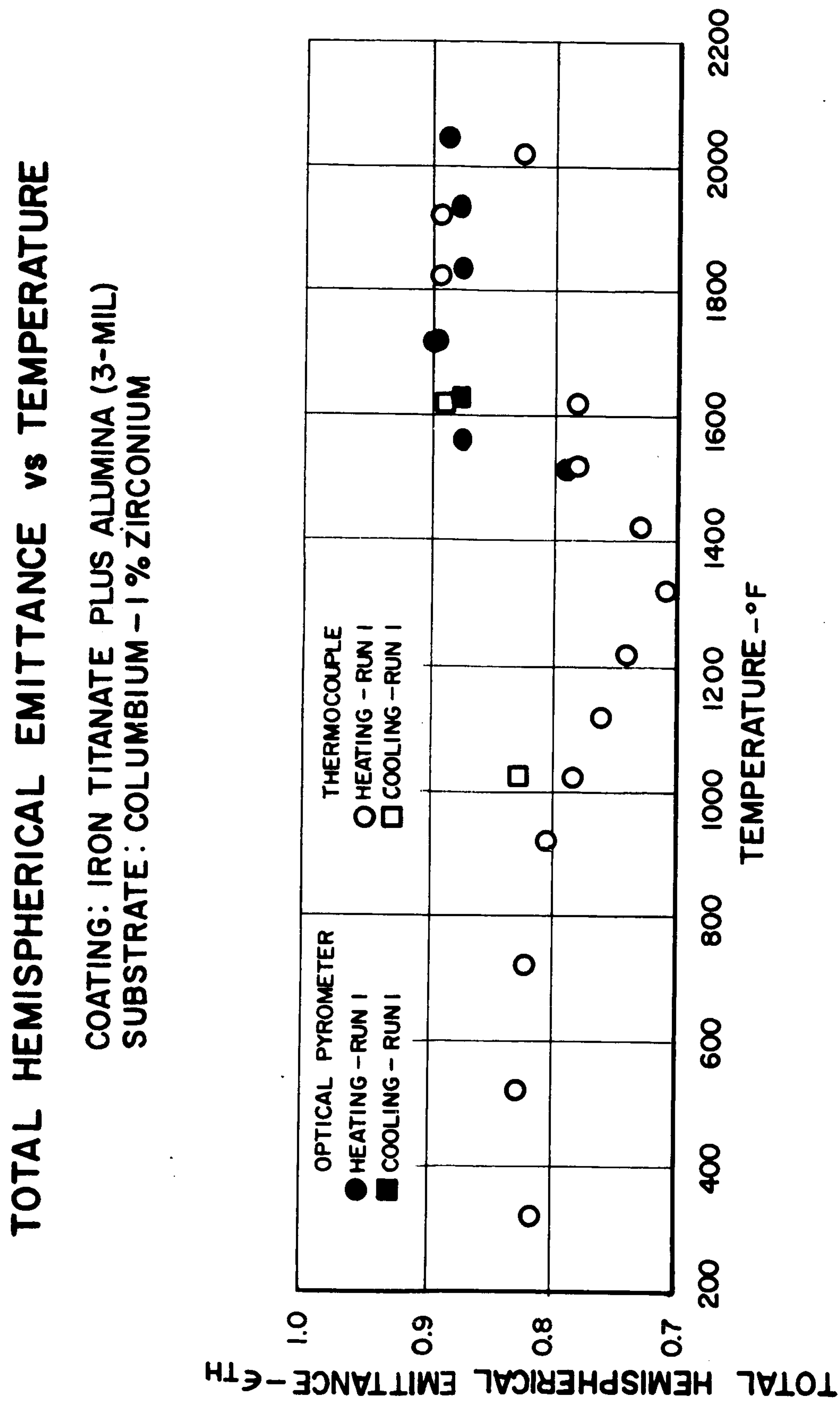

TOTAL HEMISPHERICAL EMITTANCE vs TEMPERATURE
COATING: IRON TITANATE PLUS ALUMINA (3-MIL)
SUBSTRATE: COLUMBIUM - 1% ZIRCONIUM
OPTICAL PYROMETER
HEATING - RUN I
COOLING - RUN I
THERMOCOUPLE
HEATING - RUN I
COOLING - RUN I
TOTAL HEMISPHERICAL EMITTANCE - ϵ_{TH}
1.0
0.9
0.8
0.7
200
400
600
800
1000
1200
1400
1600
1800
2000
2200
TEMPERATURE - °F

Figure 73

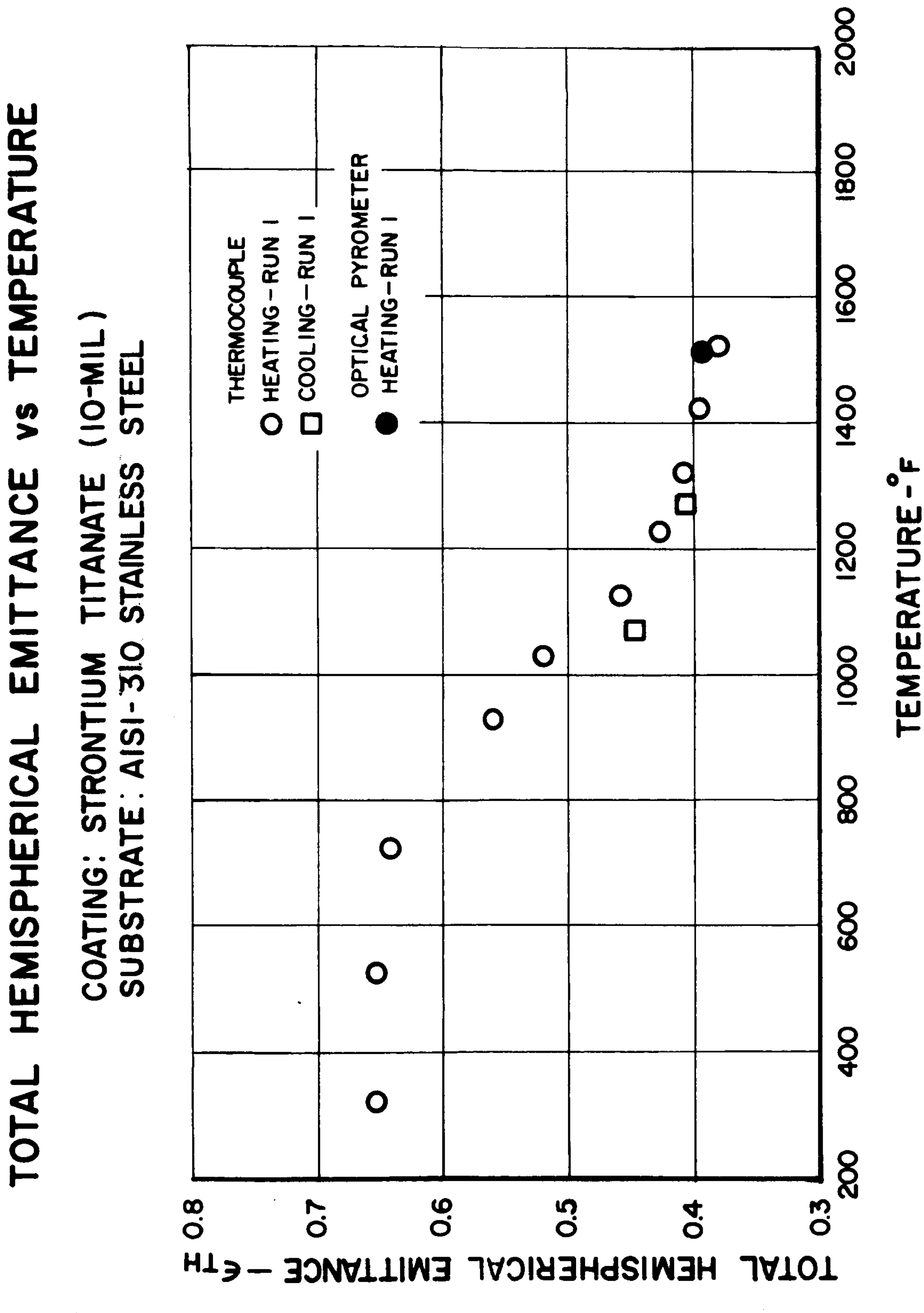

TOTAL HEMISPHERICAL EMITTANCE vs TEMPERATURE
COATING: STRONTIUM TITANATE (10-MIL)
SUBSTRATE: AISI-310 STAINLESS STEEL
THERMOCOUPLE
HEATING-RUN I
COOLING-RUN I
OPTICAL PYROMETER
HEATING-RUN I
TOTAL HEMISPHERICAL EMITTANCE — ϵTH
0.8
0.7
0.6
0.5
0.4
0.3
200
400
600
800
1000
1200
1400
1600
1800
2000
TEMPERATURE - °F

Figure 74

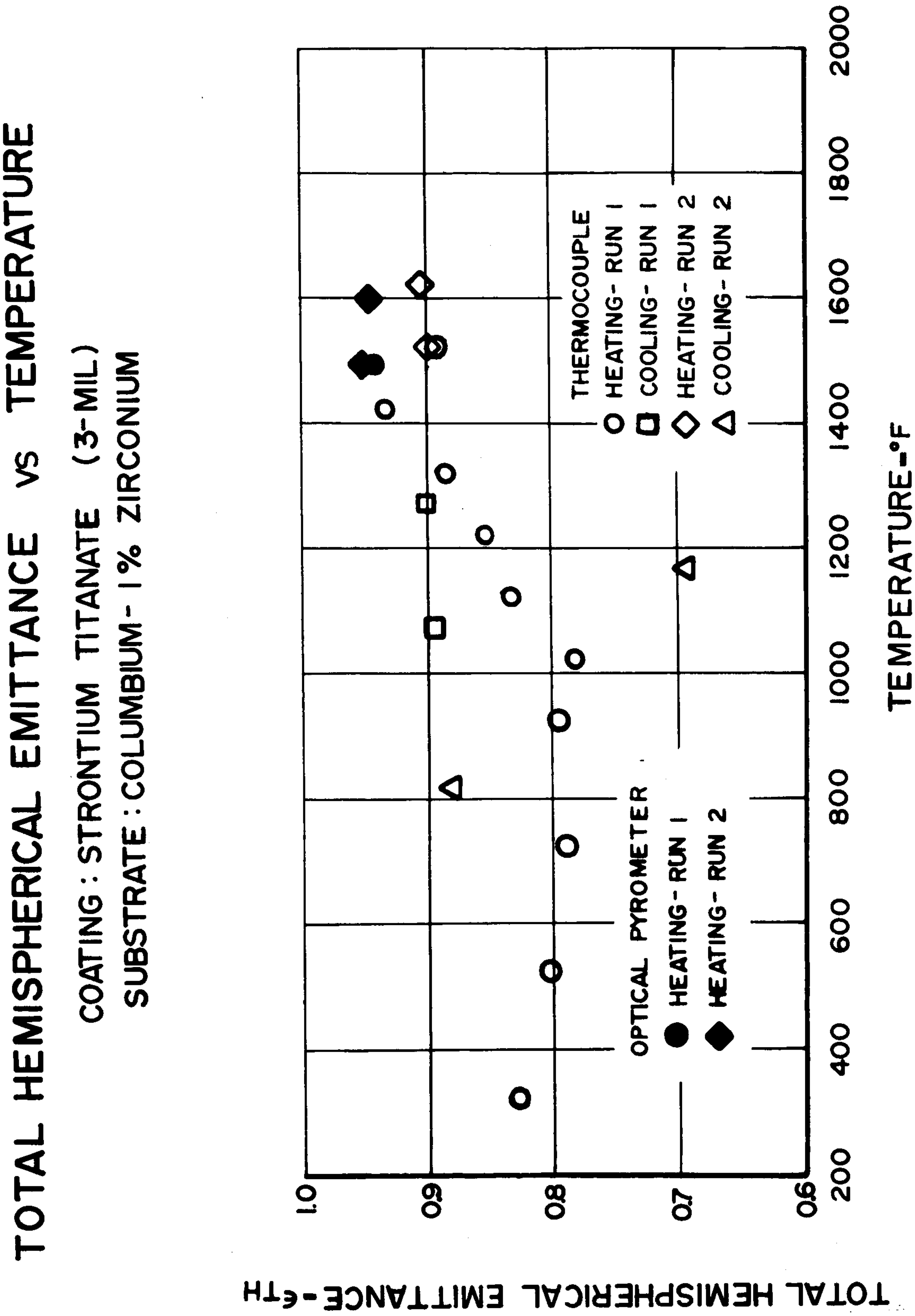

TOTAL HEMISPHERICAL EMITTANCE VS TEMPERATURE
COATING : STRONTIUM TITANATE (3-MIL)
SUBSTRATE : COLUMBIUM - 1% ZIRCONIUM
THERMOCOUPLE
HEATING - RUN 1
COOLING - RUN 1
HEATING - RUN 2
COOLING - RUN 2
OPTICAL PYROMETER
HEATING - RUN 1
HEATING - RUN 2
1.0
0.9
0.8
0.7
0.6
200
400
600
800
1000
1200
1400
1600
1800
2000
TEMPERATURE - °F
TOTAL HEMISPHERICAL EMITTANCE - εTH

Figure 75

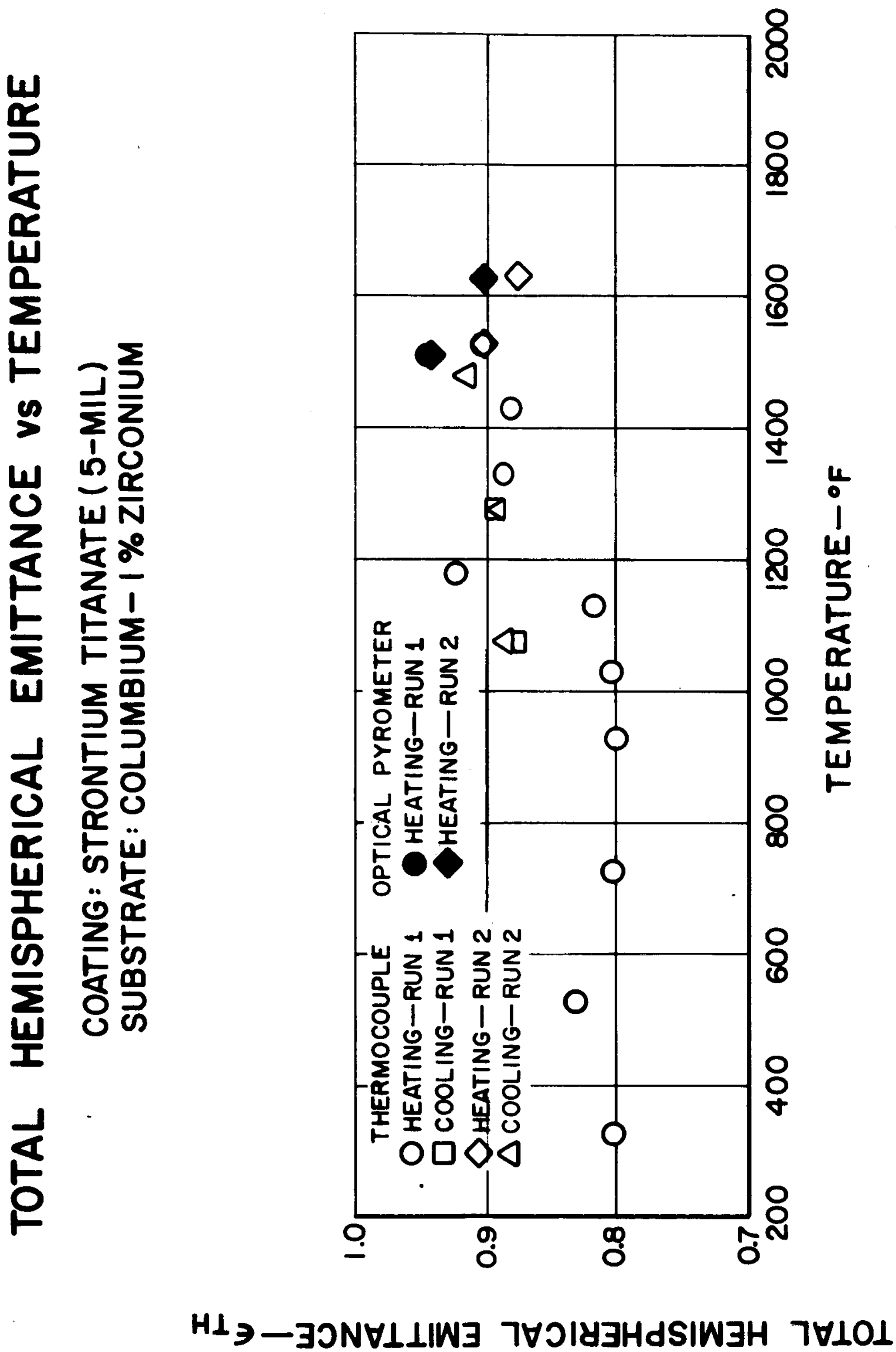

TOTAL HEMISPHERICAL EMITTANCE vs TEMPERATURE
COATING: STRONTIUM TITANATE (5-MIL)
SUBSTRATE: COLUMBIUM - 1 % ZIRCONIUM
THERMOCOUPLE
HEATING—RUN 1
COOLING—RUN 1
HEATING—RUN 2
COOLING—RUN 2
OPTICAL PYROMETER
HEATING—RUN 1
HEATING—RUN 2
TOTAL HEMISPHERICAL EMITTANCE—ϵ_{TH}
1.0
0.9
0.8
0.7
200
400
600
800
1000
1200
1400
1600
1800
2000
TEMPERATURE—°F

Figure 76

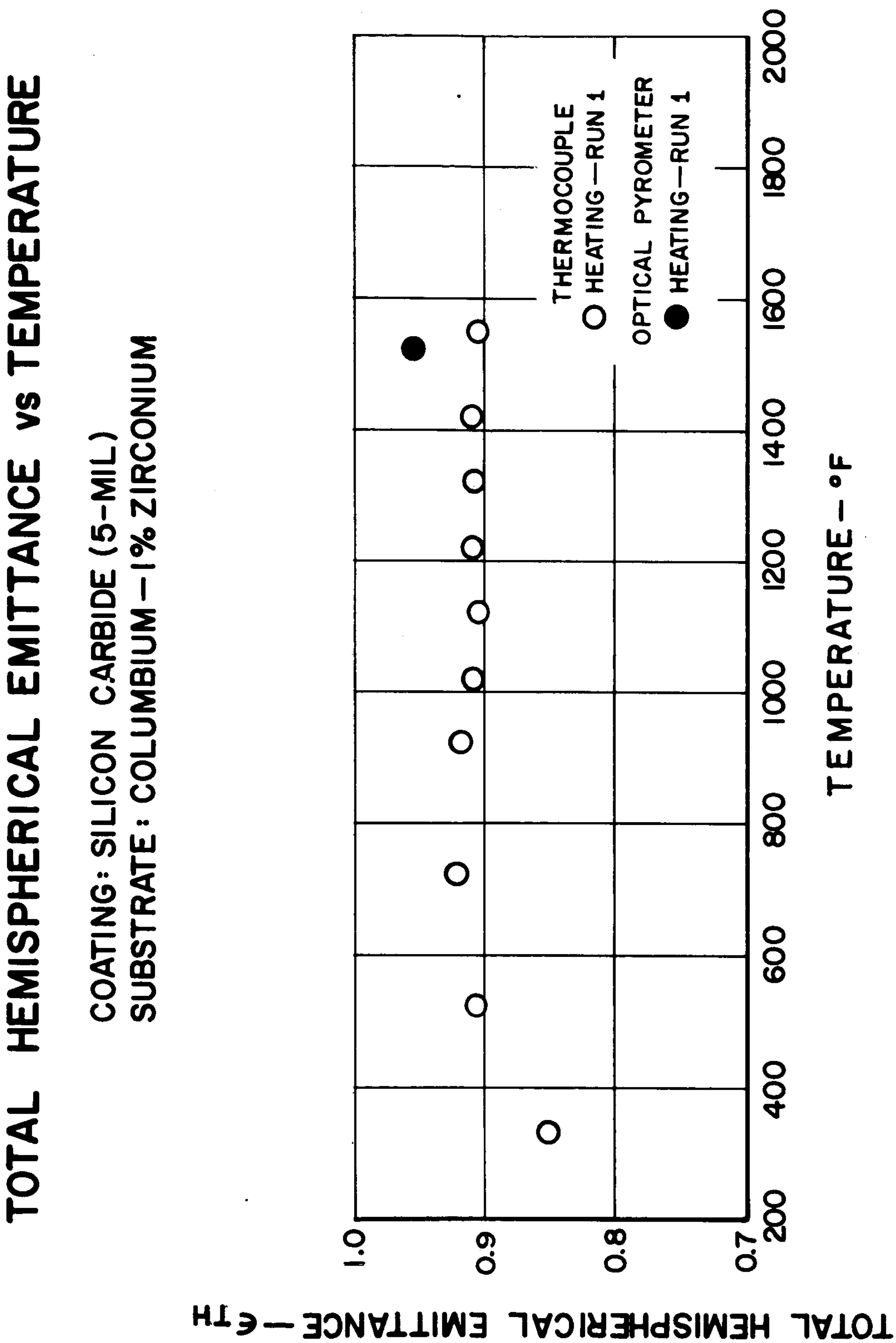

TOTAL HEMISPHERICAL EMITTANCE vs TEMPERATURE
COATING: SILICON CARBIDE (5-MIL)
SUBSTRATE: COLUMBIUM—1% ZIRCONIUM
THERMOCOUPLE
HEATING—RUN 1
OPTICAL PYROMETER
HEATING—RUN 1
TOTAL HEMISPHERICAL EMITTANCE—ϵ_{TH}
1.0
0.9
0.8
0.7
200
400
600
800
1000
1200
1400
1600
1800
2000
TEMPERATURE—°F

Figure 77

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